THE NICKEL BULLETIN

A MONTHLY SUMMARY OF CURRENT INFORMATION

JUNE, 1958

	Page
General	
Ultrasonic Welding of Nickel containing Materials	155
Nickel	
Electrorefining of Nickel: New INCO Process	155
Nickel-Cobalt Separation Extraction Process	156
Extraction of Nickel from Lateritic Ores of Cuba	157
Properties of Pure Nickel	157
Plastic Deformation of Nickel at Low Temperatures	157
Influence of Atmosphere on Creep-Rupture Properties of Nickel	157
Activation Energies for Creep of Nickel and Copper	158
Surface Analysis of Nickel Powder by Determination of Gas Adsorption	158
Dinickel-Phosphide Catalyst	158
Nickel and Nickel-Copper-Alloy Catalysts	158
Sintered-Nickel Matrix Cathode	158
Apparatus for Evaluation of Quenching Media	159
Colorimetric Estimation of Nickel in Geochemical Prospecting	159
Electrodeposition and Other Coating Methods	
Metal-Finishing Processes: Institute of Metal Finishing Conference	159
Electroplating Research by the International Nickel Company	164
Automatic Plating Plant at Vauxhall Motors, Ltd.	164
Electrodeposition of Metals on Thorium	164
'Micrograin' Nickel	165
Mechanical Finishing of Metal Surfaces	165
Non-Ferrous Alloys	
Copper-Nickel Casting Alloy for Marine Use	165
Nickel-Iron Alloys	
Analysis of Nickel-Aluminium-Cobalt-Iron Alloys	165
Cast Iron	
Purification of Cast Iron by Oxidation	165
Constructional Steels	
Dendritic Segregation of Manganese in Low-Alloy Steel	166
9 per cent. Nickel Steel for Low-Temperature Service	166
Machining of Nickel-Chromium-Steel Stub Axles	167
Filler Wires for Welding Low-Alloy Steels	168
Determination of Arsenic in Iron and Steel	168
Heat- and Corrosion-Resisting Materials	
Abnormal Grain Growth in Heat-Resisting Alloys	170
Influence of Boron and Zirconium in High-Temperature Materials	170
Alumina/Nickel Cermets	171
Centrifugal Testing of High-Temperature Materials	171
Precipitation-Hardenable Stainless-Steel Corrugated Foil for Aircraft	172
Machining of Corrosion- and Heat-Resisting Materials	172
Continuous Rolling of Stainless Steel	172
Direct Rolling of Powders	173
Reaction between Fused Sodium Hydroxide and Nickel or Nickel-base Alloys	173
Caustic Stress Corrosion of Austenitic Stainless Steel	174
Ferric Salts as Inhibitors in Acid Corrosion of Stainless Steels	174
'Monel' in Salt-Evaporation Plant	175
Analysis of Stainless Steel by X-Ray Fluorescence	175
U.S. Navy Bureau of Ships: Welding Engineers' Conference	175
Base-Metal/Interface Reactions in Brazing of High-Temperature Alloys	177

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ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

GENERAL

Apparatus for Evaluation of Quenching Media

See abstract on p. 159.

Ultrasonic Welding of Nickel-containing Materials

'Ultrasonic Welding Makes Rapid Advances.'
Steel, 1958, vol. 142, Mar. 31, pp. 80–1.

This brief article emphasizes the progress which is being made in application of the principles of ultrasonic welding to a variety of materials. A generalized diagram illustrates the main features of the process, and photomicrographs demonstrating the widening applicability of the method include examples of welding of nickel to low-carbon steel, of nickel to molybdenum, and of chromium stainless steel to titanium alloy.

NICKEL

Electrorefining of Nickel: New Inco Process

L. S. RENZONI, R. C. McQUIRE and M. V. BARKER: 'Recovery of High-Purity Sulphur, Selenium, and Cobalt in Electrorefining of Nickel.'
Amer. Inst. Mining, Metallurgical and Petroleum Engineers Annual Meeting, Preprint, Feb. 1958; 14 pp.

The history of electrorefining of nickel is closely identified with the development of the nickel-copper-bearing sulphide ores of the Sudbury district of Ontario, which, for the past fifty years, have been the major source of the world's supply of the metal. Important considerations favouring electrorefining from such ores include the inherent potential of this technique for production of metal of high purity, and the opportunity which it provides for economic recovery of precious metals as by-products.

The electrolytic refinery of the International Nickel Company, at Port Colborne, not only converts the crude nickel smelter product into metal of 99·9 per cent. purity, but also, in the course of refining, isolates such valuable by-products as copper, iron, silver, gold and the platinum metals.

Until recently, treatment prior to electrorefining has entailed sintering the crude nickel-sulphide concentrate (containing a total of 75 per cent. nickel, copper, iron and cobalt, + 25 per cent. of sulphur) to a nickel oxide having a sulphur content of only about 0·5 per cent. This sinter has then been mixed with coke and fluxes, and smelted to produce crude nickel anodes containing some 93 per cent. of nickel with 6 per cent. of metallic impurities and 0·7 per cent. of sulphur.

This paper gives a detailed account of work by The International Nickel Company of Canada, Ltd., which has culminated in development of a modified process regarded as revolutionary in the field of electrorefining. The new procedure by-passes all the traditional high-temperature operations involved in preparation of metallic nickel anodes, and employs sulphide anodes which are cast directly from the Bessemer matte. The process has the additional advantages of making possible recovery of elemental sulphur, selenium and cobalt.

The authors first briefly review the basic principles relevant to electrorefining of nickel and then describe the main stages in the introduction of improved electrorefining techniques in the Port Colborne Refinery. The original process, which employed a sulphate electrolyte, was modified in 1942 by introduction of a sulphate/chloride type of solution, which permitted the use of higher current densities, with consequent increase in plant capacity and significant saving in power. (This modification was referred to in the paper by QUENEAU, published in *Canadian Mining and Metallurgical Bulletin*, 1948, vol. 51, pp. 356–67: see abstract in *Nickel Bulletin*, 1948, vol. 21, No. 8–9, pp. 113–14.) The use of a sulphate/chloride electrolyte also laid the foundation for the recovery of cobalt as a valuable by-product, and was a factor in the sulphide-anode development.

Attention is directed to attempts which have been made by various investigators to use anodes of high sulphur content, and to the advantages which will accrue from employing sulphide anodes in electrorefining of nickel. In the INCO laboratories an interesting stage was reached in 1951, when a small piece of cast nickel sulphide was first electrolysed. This anode corroded smoothly, passing the nickel and other base metals into solution, leaving an anode sludge consisting of elemental sulphur, the precious metals, and insignificant amounts of undissolved sulphide. Tests on an anode containing 25 per cent. of sulphur revealed that the corrosion mechanism of such an anode is different from that operating in the case of a metal anode, where nickel and other metals are oxidized to the ionic state. With sulphide anodes the metals are already essentially in the ionic state, and the anodic reaction involves oxidation of sulphur, in the form of sulphide, to the

elemental state, with release of metal ions to the solution. This reaction ($Ni_3S_2 - 6e \rightarrow 3Ni^{++} + 2S$) required an increase in anode potential from the 0·2 volt, used with metal anodes, to about 1·2 volts. Anode sludge was found to contain about 95 per cent. elemental sulphur, 0·15 per cent. elemental selenium, with the remainder precious metals and sulphides. Anode-efficiency studies were made to determine the influence of composition of the electrolyte, and the sulphur content of the anode. The results indicated that, for optimum efficiency, the sulphur content should be more than 23 per cent. Copper, a major impurity in refining of nickel, was shown to exert only a minor influence on the corrosion of the sulphide anodes, and such effect was only in relation to the sulphur content necessary to avoid its selective solution. Tests with various electrolytes, as affecting anode activity, showed that (1) an all-sulphate solution was relatively unattractive on account of the low anode activity and the low sulphur content of the anode sludge; (2) an all-chloride electrolyte was less desirable than the sulphate/chloride solution.

No serious problems were encountered in producing cathodes of satisfactory purity from the sulphide anodes, but before proceeding to large-scale use it was found necessary to overcome a tendency to cracking which occurred in the early full-size anodes of that type. Rate of cooling after casting proved to be a determining factor: an account is given of the commercial casting-production procedure eventually developed.

Having established methods for satisfactory full-size-anode production, pilot-scale studies were made of anode corrosion, electrolyte purification and factors affecting the purity and appearance of the resulting cathodes. Experience so gained, together with investigations relating to tank-lining materials, cathode diaphragms and anode bags suitable for use with the sulphide anodes, are recorded in the paper.

The final section of the report is concerned with full-scale commercial production, by the new technique, of nickel (the purity of which is in every respect comparable with that of metal produced from metallic anodes), of cobalt, of the precious metals, and, for the first time on a commercial basis, of elemental sulphur and selenium. Information given in the text is supplemented, in all cases, by relevant flow sheets.

Cobalt is precipitated from the nickel electrolyte by selective oxidation with chlorine, and, after hydrolysis to cobaltic hydroxide and calcination to oxide, is either marketed in the form of oxide (cobalt content 70 per cent.) or treated further for production of electrolytic cobalt (99·55 per cent. purity).

With regard to treatment of the sulphide-anode sludge, the following details are given. Removal of elemental sulphur and selenium, by melting and filtering, leaves a filter cake containing about 50 per cent. of sulphur and all the precious and base-metal values of the sludge. The material is re-melted and cast into secondary sulphide anodes, which are electrolysed in the pilot plant. The anode sludge produced from the secondary anodes contains about 90 per cent. elemental sulphur, which is recovered by filtration. The residue, containing about 10 per cent. of precious metals, is chemically processed for recovery of a precious-metals concentrate. Separation of sulphur from selenium is effected by a distillation process which has been developed in collaboration with Blaw-Knox Company; equipment and procedure used are described in this paper.

Nickel-Cobalt Separation-Extraction Process

V. N. MACKIW, W. C. LIN, R. L. BENOIT and T. W. BENZ: 'Nickel-Cobalt Separation at Sherritt Gordon Mines Limited.'

Amer. Inst. Mining, Metallurgical and Petroleum Engineers, Preprint, Feb. 1958; 23 pp.

The paper deals specifically with the process developed at Sherritt Gordon Mines for separation and production of metallic cobalt from the mixed sulphides precipitated in the refinery operations. As a background, the following outline is given of the earlier stages of the extraction process:

'The Sherritt Gordon Refinery at Fort Saskatchewan treats sulphidic nickel-copper-cobalt concentrate in a continuous leaching operation, using ammonia and compressed air at 180°F. (82°C.) and 100 p.s.i.g. total pressure. (See F. A. FORWARD, *Jnl. of Metals*, 1955, vol. 7, Mar., pp. 457–63; *Bull. Inst. Metals*, 1954, vol. 2, pp. 13–16.) Nickel, cobalt and copper are dissolved in solution, in the form of complex metal amines. Iron remains with the undissolved solids, mainly as ferric oxide, and is separated from solution by thickening and filtration. Copper, precipitated from solution as copper sulphide, and excess free ammonia, are removed in a continuous distillation and filtration operation. A stripping step, using hydrogen sulphide or flower of sulphur, removes the residual copper, and a copper-free solution is produced.

'The copper-free solution is purified in a combined oxidation and hydrolysis operation, where the unsaturated sulphur compounds are oxidized and ammonium sulphate is hydrolyzed. The purified solution, containing 45 g./L. nickel and 0·8 g./L. cobalt, is then subjected to a batchwise reduction operation, using hydrogen as the reducing agent and ferrous sulphate as a catalyst. Nickel is reduced selectively and precipitated as a metal powder. The reduction is continued until the nickel concentration in solution decreases to approximately 0·8 g./L. At this point only about 8 per cent. of the cobalt present in solution is reduced. Accordingly, the first step in the separation of nickel and cobalt is achieved during the routine reduction operation in the nickel refinery. (See *Bull. Inst. Metals*, loc. cit., and MACKIW, LIN and KUNDA, *Trans. Amer. Inst. Mining Engineers*, 1957, vol. 209, pp. 786–93.)

'The solution, after nickel reduction, contains approximately 0·8 g./L. each of nickel and cobalt. These metals are precipitated from solution with hydrogen sulphide and the resultant mixed-sulphides residue is separated from solution by filtration.

The barren solution, containing ammonium sulphate, is sent to the crystallizer for the production of fertilizer-grade ammonium sulphate. The mixed nickel-cobalt-sulphides precipitate is then treated in a pressure-leaching and reduction operation, for recovery of both nickel and cobalt.'

The cobalt-recovery process, with which this paper is concerned, was finally developed in 1955. It consists essentially of the following operations:

(1) Preparation of the sulphide slurry for leaching.

(2) Dissolution of nickel and cobalt as sulphates, by oxidation in aqueous acid solution at elevated temperature and pressure.

(3) Removal of iron from the leach solution, by precipitation and filtration.

(4) Preferential reduction of nickel by hydrogen, at elevated temperature and pressure.

(5) Reduction of cobalt by hydrogen, also at elevated temperature and pressure.

(6) Washing, drying and briquetting of the cobalt powder.

Operations involved at each stage of the process are described.

Extraction of Nickel from Lateritic Ores of Cuba

F. WILSON: 'The Moa Bay-Port Nickel Project. New Free-World Source of Nickel and Cobalt from Cuban Ores.'

Soc. Mining Engineers, Amer. Inst. Mining Engineers, Preprint 5820*P*4, Feb. 1958; 4 pp.

Since 1951 Freeport Sulphur Company has been actively engaged in developing a new source of nickel and cobalt at Moa Bay, Cuba. Reserves in this area are estimated as amounting to slightly more than 50,000,000 tons, averaging nickel 1·36, cobalt 0·13, iron 46, per cent.

Processing of the ores will involve acid leaching to extract nickel and cobalt from the iron laterites, followed by precipitation of metallic values as a sulphide concentrate and ultimate extraction of nickel and cobalt metals by selective hydrogen reduction of sulphate solutions. The process has been fully tested in two pilot plants and commercial production is scheduled to begin in 1959.

This paper contains a short general statement on the deposits at Moa Bay, and an outline of the mining and refining processes which will be used.

Properties of Pure Nickel

MOND NICKEL CO., LTD.: 'The Properties of Pure Nickel.'

Survey prepared by *The Technical Information Section of the Development and Research Department*, Apr. 1958; 79 pp.

The survey, which covers the period 1948-1958 (March), contains data extracted from reports of original determinations only: review papers and derivative data have been omitted. (For information on properties for which no determinations have been made within the period covered, attention is directed to the publications of The Mond Nickel Company, Ltd., The International Nickel Company, Ltd., and Messrs. Henry Wiggin and Company, Ltd.) The survey includes data on physical, electrical and electrochemical properties of pure nickel.

Plastic Deformation of Nickel at Low Temperatures

P. HANSEN: 'Plastic Deformation of Nickel Single Crystals at Low Temperatures.'

Philosophical Magazine, 8*th Ser.*, 1958, vol. 3, Apr., pp. 384–418.

Single crystals of nickel (purities 99·98 and 99·4 per cent.) were deformed in tension at various temperatures between 4·2° and 300°K. (−269° and 27°C.). Shear-stress/shear-strain relations were determined, also the reversible change of flow stress with temperature. The work-hardening parameters obtained are discussed in terms of dislocation theory which has been developed mainly on the basis of data on aluminium and copper.

Influence of Atmosphere on Creep-Rupture Properties of Nickel

P. SHAHINIAN and M. R. ACHTER: 'A Comparison of the Creep-Rupture Properties of Nickel in Air and in Vacuum.'

U.S. Naval Research Laboratory Report 5036, Oct. 18, 1957; 14 pp.

This work formed part of an extensive investigation of the influence of environment on the creep properties of metals and alloys. Tests reported earlier by the senior author had confirmed published reports that the creep-rupture properties of stainless and low-alloy steel and of nickel- and cobalt-base alloys were higher in air than in a non-oxidizing atmosphere, and had indicated the rôle of stress concentration, showing that alloys of relatively low ductility had proved more susceptible to the strengthening effect of an air environment when notched specimens were used (see *Trans. Amer. Soc. Metals*, 1957, vol. 49, pp. 862–78; *Nickel Bulletin*, 1957, vol. 30, No. 1-2, p. 16). Although it was therefore evident that the high-temperature strength of metals is influenced by the type of atmosphere in which the test is carried out, the mechanism of this effect was still uncertain. The work now reported, which involved measurements of the creep rate and rupture life of nickel as a function of environment, temperature, stress, grain-size and surface notching, was carried out as a means of gaining a better fundamental understanding of the phenomenon.

Creep properties were determined, in air and *in vacuo*, at 1200° and 1500°F. (650° and 815°C.). It was found that the influence of the atmosphere was affected by the rate of strain to which the specimen was subjected. At low rates of strain the strength of the metal was higher in air than *in*

vacuo; at high strain rates the position was reversed. At very low stresses specimens tested in air at 1500°F. (815°C.) exhibited a marked increase in strength, which was attended by extensive internal oxidation and virtual cessation of creep. At high stresses the vacuum-tested specimens showed the better creep-resistance. Further, a reversal in the relative creep strength occurred during the test. In the early stages the specimen tested in air had the better creep-resistance; at a later stage there was a reversal and the vacuum-tested material had the lower creep rate. (In this connexion see comments below.)

The data obtained are compared with those derived from similar tests on a nickel-chromium alloy, 'Nichrome V'. This comparison is, however, made in the report covering that particular investigation and the conclusions drawn are similar. (See SHAHINIAN and ACHTER, *Trans. Amer. Soc. Metals*, vol. 51; abstract in *Nickel Bulletin*, 1958, vol. 31, No. 2, p. 50.) In the latter report the results for 'Nichrome V' are discussed also in relation to a mechanism which the authors then tentatively advanced to explain the effect of environment on creep. Fuller details of the mechanism are given in the present report. It is pointed out that very often reversals of rates of processes are explained by the simultaneous operation of competing effects. It is suggested that, in the cases examined, oxidation-strengthening is dominant at the stage at which low creep rates occur, whilst reduction of strength, due to lowering of surface tension, is the dominant factor when high creep rates are observed. The grounds for this theory are discussed at some length.

Activation Energies for Creep of Nickel and Copper

P. R. LANDON, J. L. LYTTON, L. A. SHEPHERD and J. E. DORN: 'The Activation Energies for Creep of Polycrystalline Copper and Nickel.'

Trans. Amer. Soc. Metals, vol. 51, *Preprint* 73; 14 pp. + 6 figures.

Activation energies for creep of polycrystalline copper and nickel were obtained by the technique involving the effect of an abrupt change in temperature on the creep rate. In general the activation energy for creep was found to increase with rising temperature, from a low value of about 3,000–5,000 calories per mole at 78°K. (−195°C.) to that for self-diffusion at the highest temperatures. The experimental results are discussed in terms of dislocation intersection, cross slip, solute-atom locking and climb mechanism for creep.

Surface Analysis of Nickel Powder by Determination of Gas Adsorption

M. W. ROBERTS and K. W. SYKES: 'Nickel Powder with Adsorptive Properties Approaching those of Evaporated Nickel Films.'

Trans. Faraday Soc., 1958, vol. 54, Apr., pp. 548–56.

Previous investigations by the authors had been concerned with the possibility of analysing the complex surfaces of reduced metals by comparing their adsorptive properties with the known behaviour of evaporated films (see *Proc. Royal Soc., Ser. A*, 1957, vol. 242, Nov. 19, pp. 534–43; *Nickel Bulletin*, 1958, vol. 31, No. 3, p. 69). For the earlier experiments the material used was nickel powder which had been reduced in hydrogen for only 12 hours. In the present paper the authors report the results of experiments in which the surface of the same nickel powder was studied (by measurement of hydrogen and krypton adsorption) throughout reduction in hydrogen at 450°C. for 102 hours.

Adsorptive behaviour closely resembling that exhibited by evaporated nickel films was observed as a result of a gradual improvement in surface impurity. This improvement is attributed to the removal of surface oxide by reduction and of non-volatile impurities by aggregation into separate phases or diffusion into the lattice.

Dinickel-Phosphide Catalyst

N. P. SWEENEY, C. S. ROHRER and O. W. BROWN: 'Dinickel Phosphide as a Heterogeneous Catalyst for the Vapour-Phase Reduction of Nitrobenzene with Hydrogen to Aniline and Water.'

Jnl. Amer. Chemical Soc., 1958, vol. 80, Feb. 20, pp. 799–800.

Dinickel phosphide, prepared by reduction of nickel orthophosphate in excess hydrogen for 15·7 hours at 545°C., was found to be a highly active heterogeneous catalyst for the reaction named.

Nickel and Nickel-Copper-Alloy Catalysts

L. E. CRATTY and W. RUSSELL: 'Nickel, Copper and Some of their Alloys as Catalysts for the Hydrogenation of Carbon Dioxide.'

Jnl. Amer. Chemical Soc., 1958, vol. 80, Feb. 20, pp. 767–73.

X-ray diffraction and magnetic measurements on nickel, copper, and alloy catalysts indicated that, although they were prepared by the reduction of oxides obtained from precipitated metal carbonates, they were essentially homogeneous equilibrium solids. Alloying 11 per cent. or less of copper with nickel caused sharp parallel drops in both the magnetic susceptibility and in the power of the catalysts to hydrogenate carbon dioxide to methane rather than carbon monoxide. Alloys containing more than 50 per cent. of copper behaved essentially like pure copper, in that carbon monoxide, but no methane, was produced.

Sintered-Nickel-Matrix Cathode

R. W. FANE: 'A Sintered Nickel-Matrix Cathode.'

Brit. Jnl. Applied Physics, 1958, vol. 9, Apr., pp. 149–53.

Oxide cathode materials, developed by Bell Telephone Laboratories, were described by MCNAIR *et al.* in 1953 (see *Jnl. Applied Physics*, vol. 24, pp. 1335–6).

These 'moulded', or 'matrix', cathodes offer the advantages of low operating temperature, combined

with favourable electrical and mechanical properties. The paper now published reports study of one variety of this type.

The composite cathode was prepared from carbonyl nickel powder and co-precipitated (Ba, Sr, Ca)CO_3. Powder-metallurgy methods were used to form the cathodes. The author discusses the specification requirements which must be met by the raw materials, and gives details of the preparation of the cathode mixture and the processing technique. Performance of the cathodes under various test conditions is reported.

The metallography of the composite material is examined, and is correlated with the characteristics of the nickel-rich end of the nickel-boron constitutional diagram.

Tests already made show that cathodes of the type described, comprising nickel, alkaline-earth carbonates and boron, can give lives of at least 8,000 hours at d.c. current densities in excess of $\frac{1}{2}$A/cm^2. Such cathodes also exhibit good pulse-emission properties. Processing technique and operating temperatures similar to those used with normal oxide-coated cathodes may be used with the matrix type of cathode.

Apparatus for Evaluation of Quenching Media

'Device for Measuring the Effectiveness of Quenching Media.'

Machinery (*Lond.*), 1958, vol. 92, Mar. 28, p. 716.

The magnetic 'Quenchometer' described in this brief note was designed by General Motors Process Development Section, Detroit, as a means of determining the quenching effectiveness of oils, water, brines and other liquids employed in the heat-treatment of steels and alloys. It is based on the fact that a magnetic material heated to a temperature above its Curie point loses its magnetism and regains it only when the temperature again falls below that point. The 1-in. (25-mm.) ball used in the test is made of high-purity nickel, which, because of its high resistance to scaling and cracking caused by thermal cycling, is an ideal material for the purpose.

The Curie point of nickel is at 670°F. (355°C.). When, therefore, prior to a test on a quenching fluid, the ball is heated to 1600°F. (871°C.), it becomes essentially non-magnetic. The heated ball is dropped into a small cage suspended in a cup of the quenching medium being tested. When the ball cools to a temperature below the Curie point it is attracted towards a magnet placed near the container, carrying the cage with it. A circuit accurately measures the time from the instant the ball drops into the cage until it is attracted to the magnet. This interval is a measure of the cooling capacity of the quenching fluid.

Ultrasonic Welding of Nickel

See abstract on p. 155.

Colorimetric Estimation of Nickel in Geochemical Prospecting

H. BLOOM: 'Rapid Field Methods for the Colorimetric Determination of Nickel for Use in Geochemical Prospecting.'

Soc. Mining Engineers, Amer. Inst. Mining Engineers, Preprint 5820*A*6, Feb. 1958; 5 pp.

Much of the recent progress in geochemical prospecting may be attributed to development of simpler and more accurate field-analysis techniques, but the authors point out limitations, from the point of view of the prospector, of the methods currently employed for determination of nickel. They describe two field procedures, one intended for measurement of 'total nickel' in soil, rock and sediment, the other for determination of 'available nickel' in soil and sediment. ('Total nickel' is defined as the amount of metal extracted from the sample by hot nitric-acid solution after digestion for one hour: 'available nickel' refers to the metal extracted by ammonium citrate at ambient temperature.) Dimethylglyoxime reacts with nickel in a buffered ammonium-citrate solution containing the detergent 'Aquet' (butyl phenoxy polyoxyethylene ethanol) and hydroxylamine-hydrochloride. The resulting nickelous dimethylglyoxime is extracted with xylene, giving colours which vary from pale yellow through orange to pink, according to the amount of nickel present. Visual comparison with standards permits estimation of 50–10,000 p.p.m. of nickel. Soil, rock or sediment may be decomposed by digestion with hot nitric-acid solution. With minor modifications of procedure, sediment and soil can be semi-quantitatively analysed for nickel on site. Full details of procedure are given.

ELECTRODEPOSITION AND OTHER COATING METHODS

Electrodeposition and Other Metal-Finishing Processes

Papers presented at the Annual Conference of the Institute of Metal Finishing, held in April, were concerned with many aspects of the surface treatment of metals. The scope of the individual papers is indicated below.

Composite Coatings: Atmospheric-Exposure Tests

B. B. KNAPP: 'The Effect of Interposed Layers of a Second Metal on the Weather-Resistance of Electrodeposited Nickel Coatings.'

Advance Copy No. 1; 27 pp.

The paper is introduced by a review of the history of nickel/chromium coatings, with some notes on the theories which have been advanced to explain the mechanism operating in the corrosion of electroplated metals. In this connexion reference is made also to the influence of copper as an underlay to nickel and nickel/chromium coatings.

The author reports results of an extensive series of atmospheric-exposure tests planned to evaluate

the effect, on the corrosion-resistance of decorative-grade nickel/chromium coatings, of interposing layers of a different metal between the two layers of nickel.

The work was carried out in three stages:

Programme 1 involved 'screening' tests consisting of exposure, in typical industrial and marine atmospheres, of composite coatings in which an intermediate layer of a different metal was used between the two nickel underlays. (Both layers of nickel were 'dull'-type, but the second layer was buffed before deposition of chromium.) The metals, selected to represent as wide a range of chemical activity as possible, comprised copper, iron, chromium, lead, tin, zinc, cobalt, manganese, silver, palladium, an iron-nickel alloy and a cobalt-tungsten alloy. The specimens were exposed in the form of plated-steel sheet, and as foils: full details are given of plating conditions, thickness of layers, etc.

Results, after one year's exposure, were evaluated in terms of appearance, pitting, and, for the foils, loss in weight. The observations made are discussed in an attempt to discover the causes of variations in corrosion-resistance and the nature of the corrosion mechanism. The behaviour of some of the composite coatings exceeded expectations: in most cases they showed corrosion-resistance superior to that of a single-layer coating of nickel. In the industrial atmosphere specimens carrying an intermediate layer of cobalt, tin, chromium, cobalt-tungsten alloy, palladium, silver and lead had better ratings than nickel; those in which the interlayer was zinc, copper, iron, or an iron-nickel alloy were inferior to nickel. In the marine atmosphere all the composites, with the exception of those containing iron, gave better corrosion-resistance than single-layer nickel. Coatings giving the best performance in both atmospheres were those having interlayers of chromium, cobalt, and cobalt-tungsten alloy. Comparison of pitting in the single-nickel-coat control specimens, with that occurring in the composites, showed that interposition of copper decreased the size, but increased the number of the pits; lead, tin and zinc increased the extent of pitting and made the pits more star-shaped; iron, palladium and silver appeared to have no effect; chromium increased the size of the pits, but lessened their number. (It is noted that heat-treatment of the composites embodying chromium reversed this effect.) Of all the interlay metals, cobalt proved most beneficial in inhibiting pitting: it reduced the pit opening to a size barely visible to the naked eye. At the other end of the scale, the specimens carrying an iron-nickel interlay were the only type which showed more perforations than the control nickel-coated specimen.

Programme 2 was concerned with study of the influence of the thickness of the intermediate layers. The tests were made on specimens carrying interlays of chromium, cobalt, tin and silver (i.e., the metals which had shown most promise in the screening tests). The thicknesses of the intermediate layers used were 10, 25, 50 and 100 micro-inches. A three-layer nickel coating (the intermediate section of which was deposited from a chloride solution) was also tested, to investigate the effect of interrupting the structure without introduction of a different metal, and a specimen carrying an intermediate layer of nickel-cobalt alloy was included. In exposure in the same conditions as those used under *Programme 1* all the composites except those incorporating silver proved superior to single-layer nickel. The results indicate that the galvanic relationship of the metals used is more important than thickness of the interlay metal. The most promising composite proved to be the one containing chromium: the benefit conferred by the chromium layer was equivalent to that of a single layer of nickel of double the thickness of the chromium. It is possible that the thickness of chromium giving best results (10 micro-inches) may coincide with minimum porosity of the chromium layer. Cobalt-containing composites were less promising, because of their tendency to form blisters. (In connexion with the behaviour of the two types of coating, the author discusses the relevant galvanic relationships of the respective metals with nickel.)

Programme 3 was particularly designed to study the weathering behaviour of composites embodying bright-nickel layers with interposed chromium or cobalt. Optimum thickness of the chromium interlay was also studied. In order to obtain satisfactory adhesion of the second nickel deposit (on the chromium) a procedure was developed which involved immersion of the chromium in an acid nickel-chloride solution sufficiently long to produce a nickel flash, thus forming a basis for deposition of the second nickel coating from a dull- or bright-plating solution.

Results of a year's exposure showed that in the industrial atmosphere the weathering behaviour of the composites incorporating bright-nickel layers was superior to that of the specimens in which dull-nickel layers were used. In marine conditions the position was reversed. *The results, in general, confirm the superior corrosion-resisting properties of composite coatings incorporating chromium;* the improvement was more marked with buffed dull-nickel than with bright-nickel coatings. Tests made on composites in which the chromium layer was deposited directly on the basis metal, followed by two nickel layers, showed that when dull nickel is used some advantage might, in certain cases, be gained from such modification. Cobalt layers were as effective with bright as with dull nickel in delaying the appearance of rust on the surface, but in this series also blistering was observed. The use of 50-50 cobalt-nickel alloy reduced the size of the blisters, but favoured the less desirable crater-type effect.

Nickel/Chromium Plating of Steam Irons

G. ARNOLD: 'The Nickel and Chromium Plating of Steel Iron Covers.'

Advance Copy No. 2; 10 pp.

The paper describes developments in the plating department of Hoover, Ltd. in connexion with the

plating of steam-iron covers, in which good surface appearance must be combined with freedom from rusting in a steamy atmosphere, over a long period of service. Experiments made with coatings deposited from two types of solution (Watts type and a proprietary bright electrolyte) showed that better finish and a higher degree of corrosion-resistance was obtained in coatings made from the Watts solution, buffed before final chromium plating.

Later, plating facilities were increased, to meet rising demand, and the plating cycle was changed to one involving copper + bright nickel + chromium. Automatic plating plant installed at that stage is described. The author discusses the economic advantages obtained from the new plant and technique, also the quality of the plated parts. Full details are given of plating sequence, evaluation of the corrosion-resistance of the coatings (by acetic-acid/salt-spray corrosion and humidity tests), and of a quenching test used to assess resistance to thermal shock.

Ageing of Bright-Nickel Solutions: Influence on Quality of Deposits

'The Properties of Bright-Nickel Electrodeposits in Relation to the Period of Service of the Plating Bath.'

J. EDWARDS: 'Part I. Properties other than Corrosion-Resistance.'

V. E. CARTER and J. EDWARDS: 'Part II. Corrosion-Resistance.'

Advance Copy No. 3; 52 pp.

Under normal conditions of operation bright-nickel solutions appear to have only a limited life: they deteriorate with use until ultimately the deposits produced are no longer acceptable. Purification is then necessary, to restore the solution to satisfactory working condition. The vital stage at which such rejuvenation becomes essential is frequently judged on somewhat inadequate grounds, mainly on the basis of the appearance of the deposits. This procedure obviously involves risk that the solution will be operated to a stage at which the physical and mechanical properties of the deposits may be deteriorating, although the unserviceability of the solution is not yet reflected in the appearance of the product. A major aim of the research reported in this paper was to establish reliable information on the influence of long service of bright solutions on the physical and mechanical properties of the deposited metals, and to estimate the extent of deterioration which might be caused by undue prolongation of use.

Initially it was necessary to develop appropriate testing methods, and subsequently to determine which of such techniques would be practically useful in large-scale control of organic bright-nickel-plating solutions. In addition, on the assumption that changes in the properties of the deposits are due to gradual accumulation of impurities in the solution, it was planned to carry out periodic analysis of solutions and of deposits made from them.

By agreement with the manufacturers, two proprietary bright-nickel solutions were selected for study: 'Super-Gleamax' and 'Efco-Udylite No. 31'. Specimens were plated with 0·002 in. (0·05 mm.) of nickel and the baths were kept in operation until a stage was reached at which purification was considered essential. The ten variables shown below were evaluated, in terms of length of life of solution: testing was carried out once or twice a week (with the exception of tests to determine the levelling action of the solution, which were made only at the beginning of the experiments).

Thickness (B.N.F. jet test and thermo-electric thickness meter

Brightness

Bright-plating Range (Hull-cell tests)

Levelling action

Throwing Power (Haring and Blum throwing-power box)

Stress (Brenner and Senderoff contractometer)

Ductility (Spiral bending test)

Microhardness

Composition of Solution

Corrosion-resistance was the subject of a separate study; see Part II of this investigation.

Conclusions

It is considered that the Hull-cell test could suitably be applied to assessment of the need for corrective additions to maintain brightness, and the tests used for estimation of stress and ductility of the deposits are also thought to be of practical value. Those used for determination of brightness, throwing power and microhardness are deemed to be of little commercial value. It is noted that ageing of the solution appeared to have no effect on the accuracy with which thickness of coating can be determined by means of the B.N.F. jet test and the thermo-electric meter.

Many of the properties proved to be unaffected by age of the solutions. Throwing power remained constant, and adhesion was, in general, good throughout. Microhardness varied erratically, but the variations were not related to the age of the solution. Other properties (brightness, stress and ductility) were deleteriously affected: details are given of the nature and extent of the effects in the respective solutions.

Attempts to determine the sources of the active impurities present in the solution were not conclusive, although the results indicate that the decomposition of addition agents is probably an important source of impurity. Consideration of the factors influencing the concentration of impurities present suggests that, in normal practice, instead of removing almost all organic material from the solution by absorption on active carbon when the electrolyte no longer functions correctly, it should be more economical to treat more frequently a portion of the solution in a separate tank, repeating that treatment as necessary.

Levelling Power of Bright-Nickel-Plating Solutions

E. BERTORELLE, I. R. BELLOBONO and C. BORDONALI: 'Bright Nickel and Levelling Power.'

Advance Copy No. 4; 20 pp.

The experiments reported are a continuation of the authors' earlier studies of the cathodic phenomena involved in electrodeposition of bright coatings from nickel-plating solutions (*6th International Mechanical Engineering Congress, Paris*, June 1956, Section 2/1). On the basis of the previous investigations, they then advanced a theory which interprets the mechanism of bright-nickel plating in terms of the formation, on the cathode surface, of a more or less complete adsorption film consisting of the organic agent present and nickel ions. It is postulated that both co-ordination and inhibitive action occur in this film: the co-ordination compound which is formed from the nickel ions may inhibit the orientation of the nickel crystals deposited, and so induce the micro-levelled distribution of crystalline seeds smaller than the wavelength of light, resulting in the appearance of brightness. In summarizing the main points of this theory, as an introduction to the present report, the authors make reference also to conflicting theories which have been put forward by other workers with regard to the brightening phenomenon.

The experiments now reported were carried out on lines similar to those followed in the earlier work, i.e., a study was made of the effect, on the nickel deposits, of adding to the electrolytes various compounds capable of forming certain complexes with the nickel ions. Results are summarized of numerous tests made to determine the characteristics of electrodeposited coatings produced from electrolytes containing optimum amounts of various types of organic brightening media, with or without 700 mg./L. of saccharin. Results relating to the series in which pyridine brighteners were used are regarded as particularly interesting from the point of view of the author's theory. The paper includes a description of a metallographic method developed for study of the levelling power of the bright-plating solutions.

Consideration of all aspects of the wide range of deposits examined leads to the conclusion that levelling probably results from a differential adsorption of addition agents in the diffusion layer on the cathode surface. This may cause a differential inhibition which would favour faster deposition in the valleys than on the peaks of the cathode profile, and the roughness of the surface is gradually reduced to such a degree that a levelled deposit is produced.

Stress in Electrodeposited Nickel

T. P. HOAR and D. J. ARROWSMITH: 'Stress in Nickel Electrodeposits.'

Advance Copy No. 5; 7 pp.

Although the addition of stress-reducing agents has been practised for many years on an *ad hoc* basis, the mechanism of stress formation, and the mode of action of stress-reducing media are at present imperfectly understood. The work described in this paper was planned to provide a more fundamental approach to the subject.

The tests were made with an unmodified Watts-type solution, and with basis solutions of that type containing varying concentrations of naphthalene-1.3.6-trisulphonic acid, naphthalene-1.5-disulphonic acid, benzene-*m*-disulphonic acid, toluene-*p*-sulphonic acid and toluene-*p*-sulphonamide. The anodes used were nickel strip.

Measurements of stress were made by means of an instrument developed by the authors, which enables readings to be taken at any stage during the growth of a deposit (see *Trans. Inst. Metal Finishing*, 1957, vol. 34, *Advance Copy No.* 8; abstract in *Nickel Bulletin*, 1957, vol. 30, No. 5, p. 71).

Curves showing variation of average 'instantaneous stress' (the stress in any small increment of thickness), as a function of the concentration of the stress-reducing agent in the electrolyte, revealed the following relationships:

(1) Instantaneous (tensile) stress usually decreases with increasing concentration of stress-inhibitor, and eventually becomes compressive.

(High tensile instantaneous stress, found in all cases in the initial stages of deposition only, was usually somewhat increased by the presence of stress-reducing agents in the solution, and was not greatly affected by temperature or by cathode current density.)

(2) The relative performance of the sulphonic derivatives, as stress-relievers, was found to be as follows (in descending order of efficacy): toluene-*p*-sulphonic acid, benzene-*m*-disulphonic acid, toluene-*p*-sulphonamide, naphthalene-1.3.6-trisulphonic acid, naphthalene-1.5-disulphonic acid.

(3) Cathode current density, within the range 0·25–1·0 amp./dm.^{2}, has little general influence on the effectivenesss of a given stress inhibitor.

(4) Rise of temperature somewhat increases the reduction of stress by toluene-*p*-sulphonamide, possibly by increasing the rate of adsorption. The results are in agreement with the hypothesis that adsorption of a stress-reducing agent is an essential stage in its action.

It is concluded that instantaneous stress, as measured during deposition, is not a completely accurate value of the final stress associated with the deposit, and that production of stress is not an 'instantaneous' process coinciding in time with the formation of the lattice. It is suggested that routine measurements should be made at standard times after deposition is complete.

The authors interpret their results by means of vacant-site and dislocation theory. Tensile stress appears to result from the formation of an oriented array of edge dislocations initiated by surface vacant sites, the overcrowded side of each dislocation being the side remote from the growing surface. Compressive stress results from stress-reducing molecules being adsorbed at surface vacant sites and so initiating edge dislocations of the reverse orientation. The ratio of the concentrations of the two

types of dislocation then determines the magnitude and sign of the stress, which thus changes continuously with the degree of adsorption.

Influence of Surface Condition of the Basis Metal on Porosity of Deposits

P. A. BROOK: 'Surface Preparation and Porosity in Electrodeposited Nickel.'

Advance Copy No. 6; 12 pp.

It is generally appreciated that to secure optimum protection of a basis metal by means of electrodeposited coatings, porosity should be at a minimum. Many of the causes of porosity in electrodeposits are known: Hammond and Hothersall have classified them as follows: (1) Uneven rate of deposition; (2) local areas of low electrical conductivity; (3) loose particles on the cathode surface originating from (*a*) the solution, (*b*) the basis metal; (4) gas bubbles; and (5) surface roughness. Porosity caused by loose particles on the surface can be prevented by thorough filtering of the solution and efficient washing of the surface and that due to gas bubbles can be reduced by wetting agents, but no relatively simple relationship between cause and cure exists in the other factors which are presumed to give rise to porosity. Little is known, for example, of the influence of the physical condition of the metal surface.

The work described was carried out to investigate what effect, if any, surface preparation of a copper basis metal would have on the porosity of a nickel coating deposited under standardized conditions. The effects of eleven surface-preparation procedures were evaluated: the preparatory techniques employed (degreasing, scouring with emery paper, electropolishing, etching and rinsing) were used in varying combination, so as to provide as wide a range of surface finishes as possible.

Porosity was assessed by an autoradiographic process in which the prepared copper basis metal was first plated with a 0·0002-in. (0·005-mm.) layer of non-active nickel and then, under identical conditions, with a 0·001-in. (0·025-mm.) deposit of radioactive nickel. Finally, the specimens were left for several days in close contact with a photographic plate. Porosity in the non-active nickel deposit was measured by counting the dark areas on the plate, which indicated the extent to which radioactivity had penetrated the pores. (It was assumed that since the grain boundaries were continuous between the non-active and active deposit, pores developed in the first layer would also be continued in the second.)

Details are given of the results, and of the statistical calculations by which they were evaluated. It is concluded that 'the porosity induced by the nature of the surface layer is affected only by changes in the gross characteristics of the surface, and that methods of surface preparation employed in this work do not influence the porosity in a deposit of the thickness used.'

It is interesting to note that neither roughening of the surface with emery paper nor the presence, in certain cases, of a film of hydrophilic basic copper phosphate affected the porosity of the deposit.

Anodic Coatings

A. W. BRACE and K. POCOCK: 'Methods of Testing Anodic Coatings on Aluminium.'

Advance Copy No. 7; 19 pp.

Growth Characteristics of Electrodeposited Coatings

H. J. PICK and J. WILCOCK: 'Some Aspects of the Growth of Electrodeposits.'

Advance Copy No. 9; 15 pp.

The authors point out that since, in most cases, electrodeposited metals must be put into service in the form in which they have been originally deposited, the microstructure formed during deposition is of vital importance in determining the properties of the plated part. It is therefore urged that a more intimate understanding of the growth phenomena occurring during electrodeposition is essential in the search for conditions which will produce the 'ideal' deposit. It is the purpose of this paper to show that 'the process of crystal growth at a cathode, under conditions not too remote from those obtaining in industrial practice, can follow certain simple, systematic patterns capable of rational analysis.'

The subject is introduced by a consideration of some literature relating to growth of crystals in other processes: (1) precipitation from supersaturated solutions, (2) solidification from the melt, (3) deposition from vapour, and (4) deposition involving a chemical reaction. It is suggested that study of crystal growth in electrodeposits can be helped by drawing on the knowledge accumulated in investigation of these related aspects of growth.

The experimental work described was concerned with electrodeposition of copper, from acid copper-sulphate solutions, on single-crystal and polycrystalline copper cathodes. A description is given of the method used for observing and photographically recording the electrodeposition process at high optical magnifications.

The results, described and illustrated, demonstrate occurrence of growth by the spreading of layers and the formation of crystallographically well-defined surface features, details of which are dependent on the crystallographic orientation of the cathode surface. Observations of growth by the self-propagating spiral-step mechanism are also reported.

Colour in Metal Finishing

C. G. NEALE: 'Colour and Styling.'

Advance Copy No. 11; 9 pp.

The paper is concerned with colour in relation to finishing of motor cars.

J. M. ADAMS: 'The Measurement of Colour.'

Advance Copy No. 12; 16 pp.

Methods used for measurement of colour are

reviewed and a description is given of some of the types of instruments which may be used for this purpose.

I. S. MOLL: 'The Selection of Coloured Pigments for Industrial Finishes.'

Preprint No. 13; 16 pp.

Electroplating Research by the International Nickel Company

'Electroplating Research at the International Nickel Company Research Laboratories.'

Plating, 1958, vol. 45, Apr., pp. 360–5.

The article gives a well-illustrated description of the facilities available in the INCO Bayonne Research Laboratories and of the nature and scope of the work carried out there.

The main activities of the electrochemical section are concerned with the evaluation of new plating processes and coatings, improvement of existing processes for electrodeposition of nickel and nickel alloys and for the plating of such materials, development of new plating processes, and study of the mechanism of corrosion of electrodeposits, the microstructure of deposits, and the mechanism of electrodeposition. Development of new applications for chemically-reduced and electrodeposited coatings of nickel and nickel alloys is also an important branch of the work.

The INCO laboratory works in close co-operation with industry, and with testing and standardizing organizations concerned with deposition of metals and the use of metallic coatings. Items of particular interest in the laboratory equipment include a pilot-scale electrodeposition plant equipped with a fully-automatic plant and a manual plating line, a particularly well-equipped bench-scale laboratory (including test equipment), apparatus for study of gases in metals, an atmospheric-corrosion test station on the roof of the laboratory, and facilities for microscopic, spectroscopic and X-ray analysis.

Among the major developments with which the Bayonne Research Laboratory has been closely associated are a hard-nickel solution widely used for reclaiming worn and mis-machined parts; an all-chloride electrolyte used for production of medium-hard deposits, and noteworthy for its high conductivity and excellent anode corrosion; methods for nickel plating with insoluble anodes; and methods for making composite metals. Many important results have been obtained also as a result of fundamental research on the mechanism of the weathering of electrodeposited nickel, and from pioneering research in the field of high-speed plating.

Automatic Plating Plant at Vauxhall Motors, Ltd.

'Automatic Plating for Automobile Components.'

Metal Industry, 1958, vol. 92, Apr. 18, pp. 313–16.

The new plating plant at Vauxhall Motors, Ltd., Luton has been constructed as part of an extensive expansion programme designed to increase the firm's annual output from 130,000 to 250,000 vehicles. This installation, which is claimed to be the largest in Europe, comprises two straight-through fully-automatic electroplating plants (one for copper, the other for nickel + chromium), two subsidiary sections for deposition of zinc, and two automatic polishing machines. The total through-put of 200 jigs per hour is stated to be equivalent to a plated-surface area of 1600 sq. ft. (145 sq. m.).

This article gives illustrated details of the facilities available in the new plant. An interesting feature of the description is a diagram showing the layout of the automatic plating and ancillary units, with correlation of the individual component parts and their use in the sequence of plating operations.

See also

'A Description of New Plant and Facilities in Operation at the Works of Vauxhall Motors, Ltd.'

Sheet Metal Industries, 1958, vol. 35, May, pp. 323–94.

This article covers all parts of the plant and operations used by Vauxhall for passenger-car-body manufacture. It also includes an introductory statement by the Managing Director, MR. P. W. COPELIN, reviewing the history of the firm's development.

Electrodeposition of Metals on Thorium

J. G. BEACH, G. R. SHAER and C. L. FAUST: 'Electroplating of Metals on Thorium.'

U.S. Atomic Energy Commission, Research and Development Report BMI-T-7, Feb. 1, 1949; 16 pp.

The report describes a method for electrodepositing adherent metal coatings directly on thorium. The key to successful coating lies in preparation of the thorium surface, for which the following treatment is recommended:

(*a*) Vapour blast or abrade, to remove scale.

(*b*) Anodic pickle in 10% (vol.) hydrochloric acid (38% HCl), with 125–250 amp./sq. ft. (13·5–27 amp./dm.^{2}) for 3–5 minutes, at room temperature.

(*c*) Pickle in 10% (vol.) sulphuric acid (96% H_2SO_4) for 3–5 minutes, at room temperature.

(*d*) Rinse in tap water.

(*e*) Electroplate.

The experiments covered electrodeposition of aluminium, beryllium, zinc, chromium, iron, nickel, tin, lead, copper, silver, indium, gold, rhodium.

The plating solution used for deposition of nickel was of the following composition:

	g./L.
$NiSO_4.7H_2O$	143
$MgSO_4.7H_2O$	75
NH_4Cl	15
H_3BO_3	15

Operated at 90°F. (32°C.)
pH 5·6.
15 amp./sq. ft. (1·6 amp./dm.^{2})
Electrolytic nickel anodes are used.

Adherence was tested by impact, filing and grinding, chisel test, bending, and cyclic heating and quenching. In most cases there was satisfactory adhesion of the coatings to the base.

'Micrograin' Nickel: New Electrodeposition Process

'New Low-Cost Nickel Coating said to be Tougher than Steel.'

Chemical Age, 1958, vol. 79, Apr. 5, p. 631.

This short note outlines the main features of a novel technique for deposition of nickel coatings, recently developed by Metachemical Processes, Ltd. The process takes advantage of the tendency of electrodeposited nickel to reproduce the grain size of the basis metal. A thin flexible sheet of very finely milled graphite particles, bonded together with a resin binder to give a graphite content of 85 per cent., is attached to a mandrel and is copper-flashed. The flash coating, adopting the surface structure of the graphite sheet, has a grain size far smaller than is obtainable by normal methods. Nickel is then electrodeposited to the requisite thickness, the composite sheet is removed from the mandrel, and separation from the graphite layer is effected by means of organic solvents. The nickel electroform which remains is then ready for bonding (with epoxy resins) into position as a protective coating.

'Micrograin' nickel can be produced (from conventional plating solutions) to a hardness controllable within the range of 120–800 (V.P.H.) and, in contrast to most nickel deposits, the coatings exhibit compressive stress. Although harder and more resistant to abrasion and impact than other types of electrodeposited metal, the 'micrograin' nickel is very flexible and can be easily bent.

The material is expected to find wide application as electroforms and, in the chemical and food-processing industries, as a lining for vessels, reactors and mixers.

Mechanical Finishing of Metal Surfaces

AMER. ELECTROPLATERS' SOC.: 'Mechanical Finishing of Metal Surfaces: Bibliography.'

Plating, 1958, vol. 45, Apr., pp. 380–4.

This (unannotated) *bibliography*, prepared under the aegis of A.E.S. Research Project 18, covers the period 1913–1956. About 350 references are listed.

NON-FERROUS ALLOYS

Nickel-Cobalt Separation-Extraction Process

See abstract on p. 156.

Activation Energies for Creep of Nickel and Copper

See abstract on p. 158.

'Monel' in Salt-Evaporation Plant

See abstract on p. 175.

Copper-Nickel Casting Alloy for Marine Use

G. L. LEE: 'New Casting Alloy Combines High Strength and Corrosion-Resistance.'

Reprint of paper from *Modern Castings*, 1957, vol. 31, May, pp. 74–79; abstracted in *Nickel Bulletin*, 1957, vol. 30, No. 7–8, p. 118.

Issued by INTERNATIONAL NICKEL CO., INC.

NICKEL-IRON ALLOYS

Analysis of Nickel-Aluminium-Cobalt-Iron Alloys

D. H. WILKINS and L. E. HIBBS: 'The Determination of Aluminium, Nickel, Cobalt, Copper and Iron in 'Alnico'.'

Analytica Chimica Acta, 1958, vol. 18, Apr., pp. 372–5.

Determination of the major constituents of 'Alnico'-type magnet alloys by classical methods involves a tedious series of operations, e.g., determination of nickel with dimethylglyoxime requires a double precipitation, and the volumetric estimation of cobalt necessitates the time-consuming plotting of the potentiometric titration curve.

The authors of this paper propose a simplified procedure based on an anion-exchange separation of the chloride complexes and subsequent determination of aluminium, nickel, copper, cobalt and iron with EDTA.

Results of determinations made by the new procedure are compared with values obtained by normal wet-analysis procedure.

CAST IRON

Purification of Cast Iron by Oxidation

R. H. T. DIXON: 'The Purification of Cast Iron by Oxidation.'

Brit. Foundryman, 1958, vol. 51, Apr., pp. 197–203.

Recent developments in cast-iron metallurgy have emphasized the importance of minor elements, particularly in the production and heat-treatment of spheroidal-graphite cast iron, where complete success often depends not only on the presence of controlled small amounts of magnesium, but also on the virtual absence of deleterious elements such as lead, titanium, arsenic, antimony, etc. These requirements necessitate the use of special base irons, which are either prepared from selected raw materials or synthesized from steel scrap, ferrosilicon and a suitable carburizer. The advantages to be gained from an economical process for the purification of pig irons of more conventional composition are obvious.

Purification of iron by oxidation of unwanted elements, and their removal as slag, is widely practised in preparation for steelmaking, and the present paper gives details of an investigation to determine the feasibility of applying similar procedures to the purification of pig iron. The potential

usefulness of three different types of process was examined. The first involves blowing a stream of oxygen on to the surface of molten iron, with or without a lime-base slag. In the second method oxygen gas is bubbled through a molten-iron bath, and the third procedure makes use of two different types of oxidizing slag.

All these processes were found to reduce the contents of some of the elements which are normally regarded as impurities in cast iron, without causing excessive loss of carbon. It is concluded that the most efficient technique is that in which the gas is bubbled through the molten metal.

CONSTRUCTIONAL STEELS

Dendritic Segregation of Manganese in Low-Alloy Nickel-Chromium-Steel Ingots

R. G. WARD: 'The Dendritic Segregation of Manganese in Steel Ingots.'

Jnl. Iron and Steel Inst., 1958, vol. 188, Apr., pp. 337–42.

Although the harmful effects of banding in steel are well recognized, relatively little study has been made of the nature and extent of the dendritic segregation which is responsible. This paper reports investigation of the micro-segregation of manganese in an unworked, sub-critically annealed, nickel-alloy steel of the following composition: nickel 4·0, chromium 1·4, molybdenum 0·1, manganese 0·40, silicon 0·35, carbon 0·13, per cent. The study was made by means of autoradiography, using the radioisotope Mn 56.

The observations made indicate that manganese is concentrated between the dendrites: maximum concentrations of 0·49 per cent., and minima of 0·33 per cent. were found in the nominally 0·40 per cent. manganese steel. High manganese was accompanied by high hardness, e.g., 243 D.P.N., compared with 161 D.P.N. at points of lower manganese content. These variations in micro-hardness are found to be associated not only with segregation of manganese, but to be influenced also by segregation of other elements.

9 per cent. Nickel Steel for Low-Temperature Service: Properties in Wrought and Cast Forms

Satisfactory behaviour at the operational temperatures involved is a major factor, but not the only requirement governing the suitability of a material of construction for low-temperature service. Several materials have the requisite low-temperature properties: not all of these can also meet other essential requirements such as moderate cost, availability in a variety of shapes and sizes, and the capacity for being formed and welded by standard commercial processes. Low-carbon 9 per cent. nickel steel (covered by *A.S.T.M. Specification A* 353) was developed specifically to meet the need for a moderately-priced material for plant used in the large-scale production of oxygen. Its application in this and other fields has in the past been limited only by its lack of availability, a condition which, since it reflected the general shortage of nickel, no longer obtains. A detailed account of the research work and tests which led up to the development of this steel was given by ARMSTRONG and BROPHY in a paper to the *American Society of Mechanical Engineers* in 1947 (see *Nickel Bulletin*, 1948, vol. 21, No. 3, pp. 39–40). Investigations which culminated in the establishment of the heat-treatment giving optimum combination of tensile strength and impact-resistance (double normalizing at 1650° and 1450°F. (900° and 790°C.) followed by stress-relieving at 1050°F. (565°C.)) were reported by BROPHY and MILLER (see abstract in *Nickel Bulletin*, 1948, vol. 21, No. 11, pp. 164–5). The papers abstracted below review data derived from tests on the steel, in the wrought and cast state, respectively, at temperatures down to −320°F. (−196°C.). Compositional limits of the material are given as: carbon 0·13 max., silicon 0·15–0·30, manganese 0·80 max, sulphur 0·040 max., phosphorus 0·035 max., nickel 8·50–9·50, per cent.

T. N. ARMSTRONG, J. H. GROSS and R. E. BRIEN: 'Properties Affecting Suitability of 9 per cent. Nickel Steel for Low-Temperature Service.'

Joint Metals Engineering Conference, Amer. Soc. Mechanical Engineers and Amer. Welding Soc., Preprint 58-*MET*-3, Apr., 1958; 9 pp.

The criterion by which the steel was evaluated during the initial development stages was its ability to meet a specification minimum energy-absorption value of 15 ft.-lb. (2·07 mkg./cm.²) when subjected, in the form of Charpy keyhole-notch specimens, to impact tests at the lowest service temperature. Evidence has since been presented, however, which casts doubt on the reliability of this criterion as a means of ensuring freedom from brittle fracture. No brittle service failure has been reported, but recently the tendency has been towards greater use of the V-notch Charpy test and the employment of different energy-absorption levels for different steels, specifications being based on fracture appearance or temperature of crack propagation. The impact values reported in this paper were therefore obtained from tests on both keyhole and V-notch specimens.

Curves derived from V-notch impact tests on plate in the fully heat-treated condition show that two inflection points occur, one at temperatures slightly below −100°F. (−73°C.), the other within the range −250° to −300°F. (−157° to −184°C.), where the curve again flattens out. Values obtained at this lower point usually exceeded 28 ft.-lb. (3·8 mkg./cm.²) and were nearly always higher than Charpy keyhole values for the steel at the same temperature. Fracture appearance of specimens tested at −320°F. (−196°C.) varied somewhat with the thickness of the plate. For 1-in. (25-mm.) plate the average

percentage of shear fracture was usually about 10 per cent. In drop-weight tests on 1-in. and ½-in. plate there was no propagation of fracture at test temperatures as low as −320°F. (−196°C.), indicating that nil-ductility transition was below this temperature.

Fatigue properties, derived from tests at normal atmospheric temperatures, on notched and unnotched specimens of the 9% nickel, were found, both at the endurance limit and at stresses within the plastic range, to be higher than for most other steels of comparable tensile strength. An endurance limit of 72,000 p.s.i. (32·0 t.s.i.; 50·6 kg./mm.²) was obtained on unnotched specimens (an endurance ratio of 62·3 per cent. at the measured level of tensile strength). For notched specimens the value was 27,000 p.s.i. (12 t.s.i.; 19 kg./mm.²) (indicating a notch-sensitivity of 2·66, closely similar to that of normalized and tempered 3½ per cent. nickel steel).

To determine the effect of cold work on low-temperature properties, V-notch impact tests were made, at +80° and −320°F. (+27° and −196°C.), on specimens elongated 5 or 10 per cent. by cold rolling: the specimens were taken transverse to the direction of rolling. Cold working was found to lower notch-impact values of the steel, but the lowest impact value was still 17 ft.-lb. (2·3 mkg./cm.²) at −320°F. on 10% cold-worked material. Stress-relief treatment at 1050°F. (565°C.), restored impact resistance to a value somewhat higher than that of unstrained material.

Tests to determine the effects of slow rates of cooling from the austenitizing temperature (such as would be encountered in dealing with heavy sections, e.g., tube, sheets and flanges) showed that the V-notch impact properties of the steel were to some extent adversely influenced, but cooling even at a rate simulating that operative at the centre of a 20-in. (50-cm.) round in still air had only a very mild effect, and at −320°F. (−196°C.) impact values of more than 15 ft.-lb. were obtained.

The remainder of the paper gives, in some detail, an account of welding experiments. No difficulty was experienced in welding the 9 per cent. nickel steel plate by the metal-arc process, but welds made with a covered electrode of about the same composition as the plate material were unsatisfactory due to cracking, and the low-temperature impact properties of the weld metal were inadequate. Attempts to develop a ferritic electrode of high nickel content proved unsuccessful. Electrodes of 25-20 chromium-nickel steel were in some respects satisfactory, but the tensile strength of the weld metal did not meet specification requirements.

Fully satisfactory results were, however, obtained by welding with a nickel-chromium-iron electrode of the 'Inconel' type containing controlled amounts of titanium, niobium and molybdenum, added to reduce sensitivity to iron dilution.* Results of low-temperature-impact, transverse-strip-tension, weld-metal-tension and bend tests, on welds produced in $\frac{5}{16}$- and ½-in. (8- and 13-mm.) plate, indicate that the development of the covered 'Inconel' electrode has successfully resolved the problem of obtaining satisfactory low-temperature properties, combined with freedom from cracking in the welds.

* 'Inco-Rod A'

See also

I. B. ELMAN and L. M. DIRAN: 'Cast 9 per cent. Nickel High-Strength Steel for Low-Temperature Service.'

Amer. Soc. Mechanical Engineers, Tech. Paper 246, Sept. 20, 1957; 6 pp. + tables and figures.

Although a considerable amount of data has been accumulated on wrought 9 per cent. nickel steel (see preceding abstract), relatively little interest has been shown in the properties of the material in cast form. The steel is, however, likely to find many applications as castings, and this paper presents information which is relevant to such use.

Nine heats of the steel were cast for use in the tests.

Impact tests were made, on double-normalized and stress-relieved specimens, at temperatures ranging from +70° to −320°F. (+21° to −196°C.), with a view to determining the effects of variation in carbon content and deoxidation practice and of addition of small amounts of molybdenum. The results are presented in tabular and graphical form.

The paper also includes data on the low-temperature thermal-expansion properties of a heat-treated 9 per cent. nickel steel containing 0·14 per cent. carbon and 0·29 per cent. molybdenum. The slope of the thermal-expansion curve is shown to be constant over the temperature range −200° to +70°F. (−129° to +21°C.). Consequently, the instantaneous and average thermal-expansion coefficient would have the same value over the range, a unique property of the 9 per cent. nickel steel which makes it possible to design low-temperature equipment to very close tolerances.

'Inco-Rod A', the electrode found suitable for welding the steel in the wrought form (see left-hand column) can also be used for welding the cast steel.

Machining of Nickel-Chromium-Steel Stub Axles

'The Production of Stub Axles for Volvo Cars.' *Machinery* (*Lond.*), 1958, vol. 92, Mar. 28, pp. 700-12.

The factory of A. B. Bolinder-Munktell (a firm associated with A. B. Volvo, Gothenburg) is one of the largest and most modern in Sweden. Its products range from Diesel engines for motor vehicles and other applications to tractors and combine harvesters. The factory is almost entirely self-contained and its facilities include a mechanized foundry with a daily capacity of 40 tons (40,640 kg.). A high standard of efficiency is maintained throughout all departments by means of closely co-ordinated time-study methods. These methods are effectively applied, for example, in the axle shop, a department concerned exclusively with the production of 'front-end' components for Volvo cars, commercial vehicles

and buses. The machinery available and the methods used are illustrated in this article by a detailed analysis of the stages involved in machining the stub-axle assembly for the Volvo PV-444/45 car. The line concerned with these components is organized to produce 400 units in each 8-hour working day.

Components of the stub-axle assemblies are machined from forgings in a nickel-chromium steel of the following composition: carbon 0·35–0·42, silicon 0·15–0·40, manganese 0·70–0·90, sulphur 0·03 max., phosphorus 0·035 max., nickel 1·10–1·40, chromium 0·70–0·90, per cent. The forgings are received fully heat-treated to give an ultimate tensile strength of 90 kg./mm². (57 tons per sq. in.) and a Brinell hardness of 269–302. The full sequence of machining operations is described, with informative illustrations.

Filler Wires for Welding Low-Alloy Steels

H. W. MISHLER, R. P. SOPHER and P. J. RIEPPEL: 'Development of Filler Wires for Welding S.A.E. 4130, 4140 and 4340 Steels.'

Welding Jnl., 1958, vol. 37, Feb., pp. 41s–8s.

The increased use of high-strength low-alloy steels in the production of aircraft components has called for development of coated electrodes and filler wires which will produce welds capable of meeting service requirements. In many cases it would be advantageous to use the inert-gas tungsten-arc and inert-gas consumable-electrode processes for welding such steels, but the use of these methods has been hampered by the inadequacy of the filler wires commercially available.

This paper reports work done at Battelle Memorial Institute, under the sponsorship of Wright Air Development Center, to develop filler wires suitable for making weldments in three commercial high-tensile steels.

The first part of the investigation covered evaluation of commercially available filler wires: part two was concerned with development of experimental compositions. The aim of the research was to obtain two filler wires suitable for each steel, one to have a composition similar to that of the basis steel, the other to be of dissimilar composition. Evaluation of commercial wires was by means of (1) screening tests, which consisted of restrained-joint tests, to determine resistance to cracking; (2) radiographs of welds, to check porosity; (3) heat-treatment tests, to determine the response of the weld deposits to standard heat-treatments used for the basis steels. Following these screening tests, the tensile and notched-bar properties of the weld deposits were determined, and, based on the results of the whole series of tests, experimental wires were devised for study.

Compositions of the plate steels, which were of aircraft quality, are shown in Table I, p. 169: the filler-metal compositions (commercial and experimental) are given in Table II, p. 169.

The test procedures used and the results obtained, are reported for the individual combinations used. The main conclusions drawn from the data obtained were as follows:

'In general, the filler wires having compositions similar to those of the basis steel are superior to those of dissimilar types.

'The S.A.E. 4130A and the Mn-Ni-Cr-Mo filler wires were best suited for welding S.A.E. 4130 steel in ¼-in. or less thickness. The response of both filler wires to heat-treatment was almost the same as that of the basis metal, although the ductility was somewhat lower than was desired. The S.A.E. 6130A filler could also be used for welding this steel, although the resulting impact properties were not so good as those of the other two weld metals used.

'The best filler wires for use with the ½-in. S.A.E. 4140 steel were the 4140A and the Mn-Ni-Cr-Mo types. The S.A.E. 4140A weld metal had good resistance to cracking, with satisfactory heat-treatment characteristics and strength properties. Its impact strengths were not so good as those obtained with other filler wires used for welding other steels, but were nearly the same as those of the base material. The Mn-Ni-Cr-Mo filler wire would be satisfactory for use at around the 150,000 p.s.i. (67 tons per sq. in.); 104 kg./mm.²) level, but at other levels there is a difference between the heat-treating characteristics of this weld metal and those of the S.A.E. 4140 steel. At the lower strength levels S.A.E. 6130 filler wire might also be used.

'The 1-in. S.A.E. 4340 (nickel-chromium-molybdenum) steel was satisfactorily welded with either the wire of similar composition or with the Mn-Ni-Cr-Mo-V filler wires. The hardness of the latter weld metal was higher than that of the base steel at the lower strength levels. The S.A.E. 4340 weld deposit showed the better impact properties at the lower strength levels, but at the higher levels the impact properties of the two weld metals were similar.'

Determination of Arsenic in Iron and Steel

BRIT. STANDARDS INSTN.: 'Methods for the Analysis of Iron and Steel. Part 38: Arsenic in Iron and Steel.'

B.S. 1121: *Part* 38: 1958. Price 3/-.

The recommended method involves the precipitation of elemental arsenic by hypophosphite reduction, followed by iodimetric determination. The method covers the range of arsenic contents found in iron and steel (including high-chromium, high-tungsten and other high-alloy steels), but is not applicable to steels containing selenium. For arsenic contents up to 0·1 per cent. reproducibility is 0·003 per cent.

The specification makes detailed recommendations with respect to reagents required, sampling, and analytical procedure, and directs attention to precautions which, under certain conditions, are necessary for accurate analysis. Reference, for example, is made to an acid mixture more suitable for dissolving chromium-nickel stainless steels than is the acid solution recommended for general use.

TABLE I

Chemical Compositions and Thicknesses of Base Plate

(*See abstract on p. 168*)

Steel	Thickness		Chemical Composition							
	in.	mm.	C %	Si %	Mn %	S %	P %	Ni %	Cr %	Mo %
S.A.E. 4130 ..	$\frac{1}{16}$–$\frac{1}{2}$	1·5–12	0·30	0·26	0·50	0·20	0·010	0·13	0·90	0·21
S.A.E. 4140 ..	$\frac{1}{2}$	12	0·41	0·27	0·85	0·021	0·014	0·19	0·99	0·21
S.A.E. 4340 ..	1	25	0·39	0·29	0·71	0·010	0·010	1·78	0·83	0·27

TABLE II

Chemical Compositions and Types of Filler Wires

(*See abstract on p. 168*)

Filler Wire	Type	Chemical Composition								
		C %	Si %	Mn %	S %	P %	Ni %	Cr %	Mo %	V %
S.A.E. 4130 ..	Commercial Wire ..	0·29	0·29	0·53	0·015	0·009	—	0·97	0·22	—
S.A.E. 4130A ..	Experimental	0·31	0·55	0·68	0·008	0·007	—	1·03	0·26	—
S.A.E. 4140 ..	Fabricated from base material	0·41	0·27	0·85	0·021	0·014	0·19	0·99	0·21	—
S.A.E. 4140A ..	Experimental	0·40	0·59	1·07	0·005	0·006	—	1·01	0·24	—
S.A.E. 4320 ..	Experimental	0·20	0·30	0·67	0·008	0·010	2·07	0·91	0·39	—
S.A.E. 4340 ..	Fabricated from base material	0·39	0·29	0·71	0·010	0·010	1·78	0·83	0·27	—
S.A.E. 6130 ..	Commercial Wire ..	0·32	0·43	0·82	0·010	0·026	—	0·98	—	0·20
S.A.E. 6130A ..	Experimental	0·31	0·54	1·08	0·007	0·007	—	0·94	—	0·18
Modified 6130 ..	Commercial Experimental Wire	0·30	0·45	0·80	0·010	0·015	—	1·00	—	0·18
Modified 6130 ..	Commercial Experimental Wire	0·35	0·45	1·00	0·020	0·020	—	1·10	—	0·18
5% Chromium Steel	Commercial Wire ..	0·06	0·12	0·44	0·025	0·017	0·19	5·06	0·49	—
Modified 6130 ..	Commercial Wire ..	0·27	1·61	1·41	0·015	0·018	1·82	0·18	0·42	—
Mn-Ni-Cr-Mo ..	Experimental	0·18	0·44	1·49	0·012	0·007	1·20	0·65	0·34	—
Mn-Ni-Cr-Mo-V ..	Experimental	0·25	0·65	1·17	0·012	0·008	1·83	1·17	0·80	0·21

HEAT- AND CORROSION-RESISTING MATERIALS

Abnormal Grain Growth in Heat-Resisting Alloys

R. F. DECKER, A. I. RUSH, A. G. DANO and J. W. FREEMAN: 'Abnormal Grain Growth in Nickel-base Heat-Resistant Alloys.'

Nat. Advisory Committee for Aeronautics, Tech. Note 4082, Dec. 1957; 70 pp.

A study of abnormal grain growth during hot-working or subsequent solution treatment was made on 'Waspaloy' (air-melted and vacuum-melted material), and on 'Inconel X-550'. A limited amount of data on 'Nimonic 80A' (obtained from another source) is included in the report.

Abnormally large grain size has been found to be associated with poor high-temperature properties, particularly with low fatigue strength and brittleness under creep-rupture conditions. Production methods have been devised to minimize growth, but the method of approach to the problem has been somewhat empirical, and the work now reported was designed to facilitate control on a sounder and more consistent basis. The main objective was the establishment of the fundamental principles governing the formation of large grains during normal conditions of heating for hot working and in the course of solution treatment, in which conditions the phenomenon had been observed. A few additional experiments were made to determine the effect of temperatures higher than those normally involved in such operations.

The programme was planned to examine induction of grain growth (1) by repeated heating and cooling, and (2) by deformation. Consideration was also given to compositional effects, and the mechanism of grain growth is discussed on the basis of the observations made by the authors and others.

The main conclusions drawn from the tests are summarized below.

Non-uniform flow of metal during hot-working operations is probably the major cause of abnormal grain growth. A factor of major importance in preventing such growth is avoidance of small critical reductions. It is essential to ensure that all parts of the metal are deformed more than 10 per cent. reduction in any one working step before re-heating. Any small reduction, particularly if it includes a strain gradient such that the critical reduction will be present, is a common source of growth. Strains arising from thermal stresses during cooling can also cause susceptibility to subsequent grain growth.

Development of susceptibility to abnormal grain growth was found to be independent of the temperature at which the deformation was imposed. Working at room temperature had the same effect, on subsequent heating, as deformation at the hot-working temperatures. Actual temperature of heating also had relatively little influence, provided that the level was sufficiently high for growth to be produced. It was confirmed that the effect occurs at the normal solution-treatment temperatures, as well as in the higher temperature ranges, and that the grains produced at the higher temperatures were not substantially larger than those resulting from treatment at normal solution temperatures.

From examination of the reactions of the respective alloys it is concluded that the influence of composition is exerted mainly by the variation in the amounts of excess phases, which restrict grain growth. A somewhat smaller grain size in vacuum-melted 'Waspaloy' than in air-melted material of the same type appeared to be due to the presence of more grain-growth restrainers, resulting from a higher carbon content. At 1950°F. (1065°C.) 'Inconel X-550' did not undergo the abnormal grain-growth which occurred in 'Waspaloy' and in 'Nimonic 80A'. At 2150°F. (1175°C.), however, which is the normal solution-treatment temperature for 'Inconel X-550', such growth was evident. It is considered that the more stable niobium compounds present in this alloy restrained growth to this higher temperature.

See also

R. F. DECKER, A. I. RUSH, A. G. DANO and J. W. FREEMAN: 'Abnormal Grain Growth in 'M-252' and 'S-816' Alloys.'

Nat. Advisory Committee for Aeronautics, Tech. Note 4084, Nov. 1957; 61 pp.

The work reported in this note was on lines exactly parallel with those followed in the investigations on 'Waspaloy' 'Inconel X-550' and 'Nimonic 80A' (see preceding abstract). The results led to precisely similar general conclusions.

With regard to the characteristics of the individual alloys, it is noted that 'M-252', due, it is assumed, to its relatively high carbide content, showed only marginal growth at 1950°F. (1065°C.). Like 'Inconel X-550', the 'S-816' alloy did not undergo abnormal growth at 1950°F. (1065°C.), but some growth occurred at 2150°F. (1177°C.), the usual solution-treatment temperature for the alloy. Growth was, however, only marginal, and temperatures of 2200°-2300°F. (1205°-1260°C.) were required to cause rapid growth.

Influence of Boron and Zirconium in High-Temperature Materials

W. J. PENNINGTON: 'Improvement in High-Temperature Alloys by Boron and Zirconium.'

Metal Progress, 1958, vol. 73, Mar., pp. 82-6.

Experiments made by DECKER, ROWE and FREEMAN, in the course of a study of the influence of melting-practice variables on the high-temperature properties of a nickel-chromium-cobalt-base alloy, demonstrated that contamination of the melt by trace amounts of boron and zirconium (viâ the furnace linings) resulted in a pronounced improvement in the creep-rupture properties. (For details see *Nat. Advisory Committee for Aeronautics, Tech. Note* 4049, June 1957; *Nickel Bulletin*, 1957, vol. 30, No. 10, p. 183.) In a subsequent paper to *the Institute of*

Metals Division of the Metallurgical Society of the American Institute of Mining Engineers (abstract in *Nickel Bulletin*, 1958, vol. 31, No. 5, p. 137), DECKER and FREEMAN, reporting the results of a microstructural investigation of the same alloy, interpreted the mechanism of this improvement in terms of the marked stabilizing effect of boron and zirconium on the grain boundaries. This paper gives details of a further investigation of the effects of zirconium and/or boron content, which was undertaken by Universal-Cyclops Steel Corporation as part of a wider programme of experiments designed to permit correlation of variation in melting procedure with the scatter occurring in the properties of different heats of vacuum-melted high-temperature alloys.

Four alloys were investigated: the nickel-base alloys 'M-252', 'Waspaloy', and 'Nimonic 90', and the cobalt-base alloy 'J-1570'. Test specimens were taken from vacuum-melted heats (1) containing no boron or zirconium; (2) containing 0·005–0·01 per cent. of boron; (3) containing 0·05–0·10 per cent. of zirconium; (4) containing boron + zirconium in any combination within the limits covered by (2) and (3). Creep-rupture tests were made at 1500°, 1600° or 1650°F. (815°, 870° or 900°C.). The temperature of test was, in each case, determined by the nature of the alloy under examination. The stresses applied varied from 20,000 to 35,000 p.s.i. (9 to 15 t.s.i.; 14 to 24·5 kg./mm.²).

The results demonstrate that both boron and zirconium have a beneficial influence on the elevated-temperature properties of these alloys, as measured by creep rate, rupture life, and elongation at rupture. Use of the two elements in conjunction with one another has a more potent effect than is obtained by the use of either singly. It was observed, also, that small amounts of boron or zirconium appreciably improved the forgeability of all the alloys investigated. Boron additions to an air-melted 'A-286' alloy improved notched-bar rupture strength, thus minimizing susceptibility to notch-brittleness.

Boron content of the alloys was determined to close accuracy by means of a specially developed technique which utilized the characteristic rose colour of the boron-circumin complex. After solution with mineral acids, and subsequent sodium-carbonate fusion of insoluble residue, the boron is isolated by distillation as methyl borate. Determination is then completed colorimetrically by photometry.

Influence of Atmosphere on Creep-Rupture Properties of Nickel

See abstract on p. 157.

Alumina/Nickel Cermets

J. W. LINDENTHAL, J. G. STRADLEY and T. S. SHEVLIN: 'Alumina-base Cermets.'
Wright Air Development Center, Tech. Report 54-173, *Pt. IV*, May 1957; 14 pp.

This report is one of a series recording the results of a comprehensive investigation into the feasibility of developing alumina-base cermets showing good resistance to impact, combined with adequate high-temperature strength and oxidation-resistance. (Abstracts of the previous parts of the report will be found in *Nickel Bulletin*, 1957, vol. 30, No. 12, p. 227.)

The experiments described form a continuation of a study of the wettability of alumina by various metals. It was hoped that wetting would be improved by pre-reacting the Al_2O_3 with a second metallic oxide which, during the firing operation, would be reduced to metal in intimate association with the alumina. It was postulated that this metal would then alloy with the metal or alloy the wettability of which was being tested, and by so doing would provide an indirect means of achieving the desired bonding between the alumina and the metallic constituent of the cermet.

Part IV of the report is therefore concerned with the effect of the presence, respectively, of chromic oxide, nickel oxide and cobaltic oxide, on the wettability of alumina by iron, nickel, 18-8 stainless steel and the cobalt-base alloy 'Haynes Stellite 31'. The compacts were fired *in vacuo* or in a hydrogen atmosphere, and cermets which appeared promising after metallographic examination were prepared in bar form and tested for strength, porosity and shrinkage.

It was found that the introduction of a second oxide to some extent facilitated the wetting of the alumina by the metal constituent. The degree to which the oxide was reduced was a function of the atmosphere and its flow rate. Specimens containing 50 vol. per cent. iron and 50 vol. per cent. oxide were found to be well bonded and almost non-porous. Good-to-excellent dispersion and reaction were also observed in the case of the nickel-containing cermets; this effect was not, however, so pronounced in cermets containing stainless steel, and was negligible for those bonded with 'Stellite 31'.

Although specimens consisting of metal, chromic oxide and alumina were successfully fired in a hydrogen atmosphere, the same result was not obtained with alumina and nickel oxide, in either a hydrogen atmosphere or *in vacuo*. Since the bars disintegrated during firing, as a result of the dissociation of the $NiAl_2O_4$, it is believed that the reduction was too severe, and that with a more suitable environment, (for example, helium) it may be possible to produce non-porous specimens.

The authors regard the results as indicative of the feasibility of producing cermets containing more than 50 vol. per cent. of metal by employing a wider sintering range and a less rigidly controlled atmosphere.

Centrifugal Testing of High-Temperature Materials

The March 1958 issue of the *Journal of Metals* contains a series of reports by American metallurgists who recently visited the Soviet Union in accordance with a plan for interchange of knowledge and discussion between scientists of the two countries. The articles describe some of the impressions gained

with regard to the work of research establishments in the U.S.S.R., the metallurgical plants, technical and scientific education, and other related aspects of ferrous and non-ferrous metallurgy.

It is reported that one of the most interesting items seen during the visit of the American metallurgists was a new type of machine for testing high-temperature alloys. This is described in the paper abstracted below.

I. I. KORNILOV: 'A High-Temperature Centrifuge for Creep, Rupture and Bend Tests.'

Jnl. of Metals, 1958, vol. 10, Mar., pp. 187–9.

In the method developed by the author, centrifugal force is applied to the test specimens, and the properties of the materials are assessed by the criteria of resistance to deformation in bending or in tension. (The apparatus used is described and illustrated.)

A large number of specimens, of similar or varying composition, can be simultaneously tested, under identical conditions, with subsequent correlation of test behaviour with composition of the materials, their physical condition, etc. The apparatus provides for testing at temperatures up to 1200°C.

The use of the centrifugal-testing technique is demonstrated by reference to determination of the properties of nickel-chromium alloys, as revealed by time-deformation curves showing test results at 800° and 1200°C. The data so obtained are discussed in relation to the nickel-chromium constitutional diagram.

Precipitation-Hardenable Stainless-Steel Corrugated Foil for Aircraft

B. MITCHELL: 'Corrugated Steel Foil Promises Lighter, Stronger Aircraft.'

Iron Age, 1958, vol. 181, Feb. 6, pp. 102–4.

'17-7 PH' stainless steel, in the fully heat-treated condition, has a yield strength at room temperature of 200,000 p.s.i. (90 t.s.i.; 140 kg./mm.^{2}). This article gives details of a technique by which this high strength may be exploited, either to decrease the structural weight of a supersonic aircraft (by as much as 25 per cent.) or to enable the structure to withstand aerodynamic heating to temperatures in the region of 750°F. (400°C.). The method was developed as the result of a three years' investigation, by the Ryan Aeronautical Company, into design of lightweight structures for supersonic aircraft.

Tests with honeycomb structures, foam, posts and closely-placed stiffeners had shown that any one of these methods satisfied strength and temperature requirements, but that the advantages were nullified by the increase in weight by which they were achieved. Since the success of the investigation depended upon developing maximum strength with minimum weight, attention was turned to the feasibility of using very thin sheet. Considerations of weight having ruled out the use of stiffeners, attention was focused on the possible use of miniature corrugations as a logical means of obtaining a strengthening effect. Development of this idea showed that for maximum strength-to-weight ratio the sheet should be less than 0·016 in. (0·4 mm.) thick. Corrugations with flat faces 0·050 in. (1·25 mm.) wide and a depth of up to 0·009 in. (0·2 mm.) were found to confer adequate resistance to buckling, under a stress of 200,000 p.s.i. (90 tons per sq. in.; 140 kg./mm.^{2}) at 700°F. (370°C.), a yield strength 25 per cent. greater than that of titanium alloys which might be used for aircraft structures.

The corrugated foil can be easily joined and assembled by spot-welding and, when welded to a thin skin of the same material, has already proved its suitability for aeronautical applications.

Machining of Corrosion- and Heat-Resisting Materials

T. S. LISTER and M. D. KINMAN: 'Machining of Corrosion- and Heat-Resistant Steels and Alloys.'

Conference on Technology of Engineering Manufacture, Paper 43, Mar. 1958; 7 pp.

In this paper the authors review the problems encountered in machining stainless steels and heat-resisting alloys (taking into account the effect of variations in the properties of the individual materials), and show how they can be overcome by correct choice of tool design, machine and technique. The information relates mainly to steels of the 13 per cent. chromium or 18-8 chromium-nickel type and to alloys of the 'Nimonic' series.

After a general discussion of tool material and design, cutting fluids, and the influence of elements (e.g., sulphur + zirconium or molybdenum) which are added to stainless steels to improve machinability, the authors make specific recommendations with respect to the precautions to be taken and the most suitable technique for turning, drilling, tapping, broaching, milling and spark-machining.

Continuous Rolling of Stainless Steel

'Continuous Rolling of Stainless Steel. A Description of New Plant Installed at Shepcote Lane Rolling Mills.'

Sheet Metal Industries, 1958, vol. 35, Apr., pp. 245–58.

When the plant started production in 1953, Shepcote Lane became the first rolling mill in Britain to apply modern techniques to the continuous rolling of wide stainless-steel strip. Time has confirmed the wisdom of equipping a completely new plant for this purpose and the increase in production made possible by the mills has gone a long way to meeting the constantly growing demand for stainless-steel sheet.

A detailed description of the Shepcote Lane mills and ancillary equipment was published in *Sheet Metal Industries*, 1953, vol. 30, Sept., pp. 709–62 (see *Nickel Bulletin*, 1953, vol. 26, No. 10, p. 172), but since that time important extensions and alterations have been made. The most significant have been those relating to the hot mill, which is now capable of handling slabs of up to 8,000 lb. (3630 kg.) weight and of producing strip up to 41½ in. (105 cm.)

width, in lengths of 350 to 400 ft. (106 to 122 m.) and gauges as low as 0·10 in. (2·5 mm.). Use of the hot Steckel principle, whereby rolling is carried out by means of coiling furnaces installed at either side of the hot-finishing mill, ensures the conservation of heat necessary for rolling the large coils.

This article gives a detailed, well illustrated description of the hot mill, an additional softening and de-scaling line, a new skin-pass mill, and modifications which have been made to the slab-grinding equipment and the strip-grinding line.

Direct Rolling of Powders

'Cold-Rolled Strip from Metal Powder.'
Engineering, 1958, vol. 185, Apr. 11, p. 472.

The concept of continuous production of strip from metal powder originated early in the present century. The capabilities and limitations of the modern processes which have emerged since that time are considered, from both the technical and economic points of view, in a paper by D. K. WORN on 'The Continuous Production of Strip by the Direct Rolling Process', presented to the Powder Metallurgy Joint Group of the Iron and Steel Institute and The Institute of Metals. This article gives brief notes on the main features of the paper which, refers, *inter alia*, to a mill designed to produce continuous cold-rolled strip from metal powders.

Reaction between Fused Sodium Hydroxide and Nickel or Nickel-base Alloys

The relatively rapid advances which, in recent years, have been made in the field of nuclear energy have aroused interest in the possibility of combining the important functions of coolant and moderator in one material. Much research has centred on molten sodium hydroxide, which not only has good heat-transfer and moderating characteristics, but also offers a wide choice of operating temperatures (melting point 604°F. (318°C.); boiling point 2534°F. (1390°C.)) together with excellent resistance to damage by radiation. Its advantages are, however, counterbalanced, if not at present outweighed, by some serious practical difficulties. Fused sodium hydroxide is extremely corrosive to most materials at 1500°F. (815°C.) and this characteristic has entailed a search for a suitably corrosion-resistant material for containers. Work on pure nickel has given encouraging results, though even nickel is, under certain temperature conditions, subject to thermal-gradient mass transfer, i.e., metal is uniformly transferred from hot to cooler surfaces of the metal. (For details of work on the resistance of nickel to corrosion by fused sodium hydroxide see LAD and SIMON, *Corrosion*, 1954, vol. 10, Dec., pp. 435-9; *Nickel Bulletin*, 1955, vol. 28, No. 2, p. 27.)

Even if the problem of mass transfer were to be overcome, however, pure nickel does not, in many applications, possess sufficient high-temperature strength to meet design requirements. The aim of subsequent investigations was therefore the development of a material stronger than nickel but possessing the same degree of resistance to corrosion by fused caustic. To this end tests were made on nickel-base and other alloys which were known to exhibit satisfactory mechanical properties at high temperature. (Abstracts of reports of corrosion tests on 'Inconel', on nickel-base alloys containing molybdenum and/or iron, and on 18-8-type stainless steels have appeared in *Nickel Bulletin*, 1958, vol. 31, No. 2, pp. 55-6.) The papers abstracted below present the results of further studies to assess the suitability of nickel and its alloys for high-temperature service in contact with sodium hydroxide.

H. T. MCHENRY and H. B. PROBST: 'Effect of Environments of Sodium Hydroxide, Air, and Argon on the Stress-Rupture Properties of Nickel at 1500°F.'
Nat. Advisory Committee for Aeronautics, Tech. Note 3987, Jan. 1958; 23 pp.

Work recorded in the literature has demonstrated that at temperatures up to 1500°F. (815°C.) mass transfer is the predominant form of corrosion to which unstressed nickel is subject in the presence of sodium hydroxide. Intergranular corrosion has been found to be insignificant. Material used for containers and piping in contact with sodium hydroxide would, however, be subjected, in a nuclear reactor, to both thermal and mechanical stresses, which might considerably accelerate attack by the caustic. The investigation reported in this paper was therefore carried out to evaluate the stress-rupture properties of commercially-pure nickel in a sodium-hydroxide environment, and to determine to what extent, if at all, corrosion by sodium hydroxide would be accelerated, or the mechanism of attack altered, by the presence of stress. Since, moreover, references in the literature indicate that the gaseous environment surrounding the piping system affects the strength of the piping, this factor also was investigated, by stress-rupture tests in air and in an inert (argon) environment, on nickel tubes containing the corrosive. The effects of oxidizing and inert environments, on the strength of the tubes, were established by additional stress-rupture tests on tubes containing no caustic but exposed to various combinations of air and argon inside and outside the tubes. All the tests, full details of which are given, were run at 1500°F. (815°C.). Since recrystallization was found to occur during heating to test temperature, all the specimens were tested in the as-received condition.

The rupture lives of the tubes proved to be closely dependent on the atmosphere both inside and surrounding them. The following conclusions are reached:

(1) The rupture strength of tubes containing sodium hydroxide and tested in air was slightly less than that of tubes tested with air outside and argon inside. It is suggested that this weakening effect of the caustic is probably due to hydroxide attack on the nickel oxide present in the grain boundaries.

(2) Stress-rupture curves for tubes tested in an argon atmosphere, with and without exposure to sodium

hydroxide, show that their strength at the test temperature is not appreciably affected by contact with the caustic. Attack did not appear to be intergranular and the degree of mass transfer of nickel was only similar to that observed in unstressed tubes.

(3) Comparison of curves relating to tubes tested wholly in air with those obtained from tubes tested wholly in argon, showed that at 1500°F. (815°C.) the stress-rupture lives were much longer in air than in argon. This strengthening effect of air becomes more pronounced with increasing time-to-rupture: it is attributed to intergranular oxidation (which is, however, accompanied by severe embrittlement).

(4) It is finally concluded that at 1500°F. (815°C.) the application of stress to nickel tubes appears neither to increase the rate of mass transfer of nickel, nor to induce intergranular corrosion.

See also

H. B. PROBST, C. E. MAY and H. T. McHENRY: 'Corrosion-Resistance of Nickel Alloys in Molten Sodium Hydroxide.'

Nat. Advisory Committee for Aeronautics, Tech. Note 4157, Jan. 1958; 26 pp.

Previous investigation of the resistance of commercial high-nickel alloys (e.g., of the 'Hastelloy', 'Monel', 'Inconel' and 'Nimonic' series) to attack by molten sodium hydroxide had revealed that all these materials were much less corrosion-resistant than pure nickel. In most cases, corrosion was in the form of severe intergranular attack. The tests reported in the present paper were carried out to study the behaviour of some nickel-base alloys which, although less complex, would still be expected to display greater high-temperature strength than pure nickel.

The materials selected for testing were of two general classes:

(1) *Solid solutions*, which were studied with the aim of determining whether homogeneous single-phase alloys might be free from the severe intergranular attack observed in the commercial alloys, in which possibly the attack may be favoured by concentration of alloying elements or second phases at the grain boundaries. The binary nickel-base alloys investigated contained, respectively, copper 30, manganese 15, molybdenum 5, silicon 1, tin 5, zirconium 1, niobium 15, wt. per cent.

(2) *Two-phase materials*, on which, it was hoped, satisfactory resistance to sodium hydroxide would be conferred by the presence of small discrete particles of a second strengthening phase, mechanically distributed throughout a corrosion-resistant matrix. It was reasoned that in this way attack should largely be confined to the surface in contact with the caustic, and selective leaching of particles below the surface would be inhibited by the intervening matrix. Such structures may be produced either by precipitation-hardening or, using powder-metallurgy methods, by mechanical dispersion of refractory particles in nickel. In the first category a nickel-base alloy containing 11 per cent. of titanium was tested. In the second the materials chosen for study consisted of nickel-base masses containing respectively (wt. per cent.): titanium carbide, 12·0 or 8·2; aluminium oxide, 3·9; magnesium oxide, 4·3.

The conclusions drawn by the authors from the results of static corrosion tests in sodium hydroxide at 1500° and 1700°F. (815° and 926°C.) are summarized below. The data obtained proved most encouraging, by comparison with the disappointing results of tests on commercial alloys.

Of eleven materials evaluated, only two of nickel base (the nickel-zirconium and the nickel-titanium) showed signs of significant intergranular corrosion. Other types of attack were, however, prevalent in some of the materials, the most common being leaching of the solute, and formation of foreign non-metallic phases within the alloy. Corrosion of the materials was, in general, only slightly greater at 1700° than at 1500°F.

The alloy containing 30 per cent. copper (i.e., with the same nominal composition as 'Monel') alone exhibited corrosion-resistance comparable to that of pure nickel, but was susceptible also to the same thermal-gradient mass transfer. The nickel-molybdenum, nickel-zirconium, and nickel-tin solid solutions, and all the materials having refractory particles, are regarded as worthy of further investigation since their over-all corrosion-resistance was good.

Corrosion of the remaining solid solutions (nickel-niobium, nickel-manganese and nickel-silicon) and of the nickel-titanium precipitation-hardenable alloy was sufficiently severe to warrant their rejection as subjects for further study.

Caustic Stress Corrosion of Austenitic Stainless Steel

The following abstract is published of a paper presented to The Electrochemical Society, Apr. 1958.

G. C. WHEELER and E. HOWELLS: 'Caustic Stress Corrosion of Type 347 Stainless Steel.'

Jnl. Electrochemical Soc., 1958, vol. 105, Mar., p. 50C.

'Stress-corrosion tests of Type 347 chromium-nickel stainless steel were made in mixtures of caustic-in-water and caustic-in-sodium, at temperatures between 448° and 850°F. (231° and 454°C.), using various types of specimen. Transgranular cracking of the specimens occurred in both solvents, over a wide range of temperatures and concentrations. In miniature-boiler tests cracking was prevented by adding disodium phosphate to the boiler water at the rate of 1 part PO_4 to 4 parts caustic anion.'

If and when this paper is published in full, notification will be made in *The Nickel Bulletin*.

Ferric Salts as Inhibitors in Acid Corrosion of Stainless Steels

M. A. STREICHER: 'Corrosion of Stainless Steels in Boiling Acids and its Supression by Ferric Salts.'

Corrosion, 1958, vol. 14, Feb., pp. 59t-70t.

Variations in attack on 18-8 chromium-nickel steels exposed, under varying conditions, in boiling

oxalic-acid solutions, indicated that the presence, in such a solution, of corrosion products resulting from specimens previously exposed in it could have an inhibiting effect on the corrosion suffered by new specimens. Investigation of this phenomenon, by means of tests in solutions of oxalic acid to which various ferric compounds were added, demonstrated that such substances had a pronounced effect. The report presented in this paper relates to work arising from such observations.

The steels used in the tests included typical 18-8 chromium-nickel compositions, straight-chromium steels and high-manganese chromium-nickel-manganese types. A detailed study was made of the influence of the various alloying elements present, on corrosive attack by oxalic, formic, acetic, hydroxyacetic, phosphoric, sulphuric, and sulphamic acids and sodium bisulphate. The work included study of the influence of acid concentration, the effects of corrosion products and the presence of oxygen.

The very extensive tests made show that soluble ferric salts, present in amounts exceeding a certain critical concentration, inhibit corrosion of stainless steels, in the boiling acids used, to such a degree that the characteristic bright finish of the steels is maintained for quite long periods. The minimum amount of ferric ions required to secure protection is dependent on the alloy content of the steel (chromium, nickel, manganese). The corrosion characteristics of the high-manganese stainless steel were found, in the conditions used in these tests, to be closely similar to those of standard 18-8 chromium-nickel steels. The mechanism of the inhibiting effect of the ferric ions is discussed.

Copper-Nickel Casting Alloy for Marine Use

See abstract on p. 165.

'Monel' in Salt-Evaporation Plant

R. PETER: 'Salt-Works Engineering.'

Escher Wyss News, 1957, vol. 30, No. 2, pp. 33-42.

The firm of Escher Wyss, Zürich, has been active in the field of saltworks engineering for almost 40 years. This article reviews the main features of the firm's projects, from the first pilot plant installed at Reichenhall, in 1921, to the development of much improved equipment which is representative of that in operation at various European salt-producing plants.

Brine-purification and evaporation processes are described and details (supplemented with diagrams and flow sheets) are given of the evaporation plant.

Operational conditions in the evaporator are highly conducive to erosion, since the brine is saturated and practically at boiling point. In units built by Escher Wyss advantage is taken of the well known resistance of 'Monel' to corrosion by brine: this alloy has been shown by long experience to be ideal as a cladding material for the more exposed parts of the condenser: for example, the central downtake and the propeller.

Analysis of Stainless Steel by X-Ray Fluorescence

L. SILVERMAN and W. W. HOUK: 'Determination of Iron, Chromium and Nickel by X-Ray Fluorescence. Aqueous Solution Method.'

Atomics International Division of North American Aviation, Inc., Report NAA.SR. 1788, Mar. 15, 1957; 12 pp.

Methods for analysis of stainless steel by X-ray fluorescence usually employ solid samples of prescribed surface area and surface finish. The procedures used for such specimens are not, however, suitable for analysis of chips, drillings, or powders. This paper proposes an aqueous-solution X-ray fluorescence method which can be used for such samples.

Determination of Arsenic in Iron and Steel

See abstract on p. 168.

U.S. Navy Bureau of Ships: Welding Engineers' Conference

U.S. NAVY BUREAU OF SHIPS: 'Welding Engineers' Conference at United States Naval Research Laboratory, 30 Apr.-4 May, 1956.'

Published by the *U.S. Navy Bureau*, Sept. 1, 1956; 392 pp.

Papers presented at this conference dealt with welding equipment and technique and with specialized procedures suitable for the welding of individual materials. The scope of contributions relative to nickel or nickel-containing materials is indicated below.

Welding of Stainless Steels

R. D. WYLIE: 'Some Considerations in the Welding of Austenitic Chromium-Nickel Stainless Steels,' pp. 93-102.

Improvements in design and the greatly increased efficiency which has been achieved in steam-power generators during the past decade have of necessity resulted in more severe conditions of service: temperatures, for example, of the order of 1200°F. (650°C.) and pressures of 5000 p.s.i. (2·2 t.s.i.; 3·5 kg./mm.^{2}). The high-temperature strength and corrosion-resistance of the chromium-nickel austenitic steels make them particularly suitable for such applications, but their use has not been without its problems. Fabrication, by welding, of such steels has sometimes, especially in heavy sections, given rise to difficulties either during actual welding, during post-weld heat-treatment, or in subsequent service at elevated temperatures. This paper reviews the comprehensive programme of investigations on welding which, since 1949, has been in progress in the laboratories of The Babcock and Wilcox Company, with the particular aim of improving the high-temperature properties of the weldments.

On the basis of chronology and subject, the review may be divided as follows:

(1) A summary is made of the results of a test programme, initiated in 1949, to evaluate the high-temperature characteristics of weld deposits made from commercially available austenitic electrodes. Details of the results have been previously published by WYLIE, COREY and LEYDA (*Trans. Amer. Soc. Mechanical Engineers*, 1954, vol. 76, Oct., pp. 1093–1106: see abstract in *Nickel Bulletin*, 1955, vol. 28, No. 1, pp. 13–15). A résumé is presented of the influence, on the high-temperature behaviour of the weld deposit, of the following composition variables: silicon, carbon/silicon ratio, manganese, chromium, nickel, niobium, molybdenum.

(2) On the basis of these screening tests, a further development programme was inaugurated, in an effort to develop an electrode which would ensure the optimum combination of high-temperature strength, hot ductility and structural stability in the weld. The investigation, details of which are given here, centred mainly on the welding of the molybdenum-containing or niobium-stabilized chromium-nickel stainless steels (A.I.S.I. Types 316 and 347). This work resulted in development of a modified electrode ('Croloy 16-8-2') containing chromium 16, nickel 8, molybdenum 2, per cent., which gave welds with much improved high-temperature properties. The development of the 16-8-2 electrode was described by CARPENTER and WYLIE in *Metal Progress*, 1956, vol. 70, Nov., pp. 65–73 (*Nickel Bulletin*, 1957, vol. 30, No. 4, p. 65).

(3) In this section the six types of cracking which may result from use of restraint during welding are discussed. The theory is advanced that stress-relief is superfluous where either the weld metal, the basis material, or both, are capable of deforming at operating temperatures; the high stresses will be dissipated by deformation and no premature failure will occur. Stress-relief may not therefore be the best procedure where there is difficulty in carrying it out uniformly.

In an addendum to the paper reference is made to two development contracts at present in progress at The Babcock and Wilcox Company. The first covers the development of a basis material of the same general composition as that of the 16-8-2 electrode, which would combine the high-temperature strength of the 18-8-type stainless steels with the excellent resistance to cracking of the electrode. Data so far available on the high-temperature properties and susceptibility to cracking of bar stock of the 16-8-2 type have been encouraging, and further tests are being carried out on tubing made from the same material. The second contract, which was concerned with improving the properties of straight-chromium stainless steels, has culminated in the development of a steel which is essentially an A.I.S.I. 430 type modified by addition of 1 per cent. of nickel. Tests on this 16-1 chromium-nickel steel indicate that, with the development of a satisfactory electrode, the weld metal will have strength and as-welded ductility superior to those of any of the present range of ferritic or martensitic stainless steels. It is also claimed that the steel (up to a thickness of 1 in.; 25·4 mm.) can be normalized and tempered to produce adequate impact strength at room temperature and at 10°F. (-12°C.); and that, in contrast to Type 430 steel, no embrittlement occurs as a result of ageing at 885°F. (475°C.).

Welding of 'Ni-O-Nel'

L. ROBBINS: 'Report on the Weldability of 'Ni-O-Nel', *ibid.*, pp. 263–72.

'Ni-O-Nel', a high-nickel alloy containing chromium and iron, was developed for applications involving corrosive conditions too severe for the '18-8'-type chromium-nickel stainless steels. Typical of such conditions are those imposed by contact with Diesel exhaust gases and their condensates in the snorkel exhaust-piping systems of submarines.

This paper presents the results of tests made to determine the weldability and formability of 'Ni-O-Nel' for such purposes. Tensile, bend, hardness and impact tests were made on basis plate in the thicknesses proposed for production work, and on butt and fillet welds made by three processes: the shielded metal-arc, the inert-gas consumable-electrode and the inert-gas tungsten-arc. The corrosion-resistance of the as-welded material was also established.

The conclusions drawn from the results are summarized below.

Tensile and bend tests on the weldments showed that, with the exception of low elongation in one specimen welded by the tungsten-arc process, the values obtained satisfied the design-stress requirements for the snorkel system. Tensile and impact properties of welds made with 'Ni-O-Nel' electrodes and rods indicated that, by using a stringer-bead technique and avoiding weaving, puddling and excessive amperages, it is possible to make sound crack-free welds. It is recommended that welding in positions other than down-hand be limited to use of electrodes of $\frac{1}{8}$ in. (3 mm.) or smaller. Penetration of welds into the backing-plate root face is adequately effected by use of joint designs which give proper access and of groove angles greater than those normally used for steel. Machine welding by the consumable-electrode process is considered satisfactory, provided that the current densities employed are commensurate with the travel speeds used.

It was found that 'Ni-O-Nel' could be formed at low temperatures (i.e., within the carbide-precipitation range) followed by solution-treatment. Straightening should preferably be accomplished without heating, but heating up to a temperature below the carbide-precipitation range is permissible.

Tests by the Strauss acidified-copper-sulphate procedure demonstrated that the corrosion-resistance of the weld and the heat-affected zone, in the as-welded condition, is not significantly affected if heat input and interpass temperature are adequately controlled.

Welding of Nickel-Aluminium Bronze

J. L. CAHILL: 'Welding of Nickel-Aluminium Bronze', *ibid.*, pp. 315–18.

This is the original version of a paper the revised form of which was published in *Welding Jnl.*, 1956, vol. 35, Dec., pp. 1226–30; see abstract in *Nickel Bulletin*, 1957, vol. 30, No. 3, p. 39.

A *reprint* has been issued by INTERNATIONAL NICKEL CO. INC.

Cladding by Inert-Gas Metal-Arc Welding

L. ROBBINS: 'Cladding Operations', *ibid.*, pp. 319–31.

This paper represents the first of a projected series of reports giving details of a test programme covering investigation of the feasibility of cladding steel surfaces with corrosion-resistant weld metal by means of the multi-arc automatic inert-gas metal-arc welding process. It is anticipated that this technique would provide a means, for example, of applying a corrosion-resistant surface to torpedo-tube barrels, propulsion shafting, high-strength cast heat-treated propellors, pressure vessels, etc.

This first report covers the results of bend and tensile tests on specimens removed from high-tensile steel plates clad, respectively, with extra-low-carbon 18-8-type chromium-nickel steel containing molybdenum (A.I.S.I. Type 316L); a steel containing chromium 22-24, nickel 12-15, per cent. (Type 309); aluminium bronze and 'Monel'. The welding equipment used is briefly described and full details of welding procedure are given for each thickness of cladding ($\frac{1}{8}$ and $\frac{1}{4}$ in.; 3 and 6·5 mm.) and basis metal ($\frac{1}{4}$ and $1\frac{1}{2}$ in.; 6·5 and 38 mm.).

The author concludes that the process can be successfully used to clad plate $\frac{1}{4}$ in. thick and above. The results of tests on the stainless steels are considered satisfactory, as are those of aluminium bronze and 'Monel', provided that care is taken to maintain a controlled interpass temperature of approximately 300°F. (150°C.). This helps to inhibit intercrystalline penetration of weld metal into the prime plate, and, with 'Monel', tends to prevent hot-short cracking. Since it is essential that porosity should be at a minimum in such cladding, it is recommended that the soundness of the cladding be checked by non-destructive test methods.

Base-Metal/Interface Reactions in Brazing of High-Temperature Alloys

W. FEDUSKA: 'The Nature of High-Temperature Brazing-Alloy/Base-Metal Interface Reactions.'
Welding Jnl., 1958, vol. 37, Feb., pp. 62s–73s.

The paper reports one stage of a series of fundamental investigations in progress by Westinghouse Electric Corporation, initiated with the aim of obtaining a clearer understanding of the factors controlling the formation of brazed joints, and of adapting such knowledge to practical problems arising in the joining of metals by this method.

In this section of the research a study was made of the microstructural and hardness characteristics of a variety of high-temperature brazing-alloy/base-metal interfaces. These microstructures reveal products resulting from different reactions during brazing, and thus explain a number of the specific changes which occur during the operation.

The base metals used and the brazing alloys employed are shown in Table I, p. 178.

The textual discussion of the observations made is supported by an extensive series of photomicrographs illustrating the interfaces and the immediately adjacent metal. The following summary is made of the nature of the reactions found to have occurred in brazing the respective materials:

1. *Martensitic Stainless Steel (Type 410)*

Incipient volume-diffusion reactions generally predominated.

Below a brazing temperature of 2070°F. (1132°C.) restricted volume diffusion of brazing-alloy elements produced a complex solid solution at the joint-interface region.

Carburization of the base metal occurred by interstitial diffusion when the brazing alloy was high in carbon (e.g., Alloy 1).

At about 2070°F. (1132°C.) an unstable solid solution was formed in the interface base metal; this decomposed on cooling from the brazing temperature. The transformation produced a complex solid solution (Fe-Cr-Ni-Si) in a eutectoid matrix of complex solid solution and intermetallic compounds, which, according to the composition of the brazing alloy, was borocarbide or boride.

At brazing temperatures of 2150°F. (1176°C.) and above, incipient volume diffusion of austenite-forming nickel produced an austenite interface structure which transformed to martensite on rapidly cooling from the brazing temperature. A duplex base-metal structure, consisting of martensite islands in a ferritic matrix, was produced by heating the martensitic steel into a two-phase austenite-ferrite region at these brazing temperatures, followed by rapid cooling.

2. *Austenitic High-Temperature Alloys*

(*a*) Grain-boundary and incipient volume-diffusion mechanisms predominated in the brazing reactions reported. Gross volume diffusion was relatively insignificant under the short-time brazing conditions used.

Base-metal grain-boundary penetration occurred primarily during heating to brazing temperature: incipient volume diffusion took place at brazing temperature.

Interstitial elements, (e.g., carbon), and sometimes elements of relatively small atomic radii (e.g., boron and silicon) in the brazing alloy diffused along base-metal grain-boundary 'short-circuit' paths during heating to the brazing temperature.

Grain-boundary diffusion resulted in formation of solid solutions and/or intermetallic compounds in the grain-boundary regions.

TABLE I

Compositions and Treatment Temperatures of High-Temperature Base Metals and Brazing Alloys used in the Wettability Tests

(*See abstract on p. 177*)

Base Metals Type and/or Designation	Nominal Composition												100-Hour Ageing Temperature
	C %	Si %	Mn %	Cr %	Ni %	Co %	Mo %	W %	NbTa %	Ti %	Al %	Fe %	
A.I.S.I. 410; Martensitic Stainless Steel	0·15 max.	—	—	11·5-13·5	—	—	—	—	—	—	—	Bal.	None
A.I.S.I. 347; Austenitic Stainless Steel	0·08 max.	—	—	17·0-19·0	9·0-13·0	—	—	—	0·80 min.	—	—	Bal.	1290°F. (698°C.)
Iron-base, High-temperature Alloy, titanium hardened	0·03	0·8	0·8	13·5	26·0	—	3·0	—	—	1·6	0·1	Bal.	1290°F. (698°C.)
Nickel-base, High-temperature Alloy, titanium-hardened	0·04	0·3	0·7	15·0	Bal.	—	—	—	0·9	2·5	0·9	7·0	1470°F. (798°C.)
Cobalt-base High-temperature Alloy ..	0·10	0·7	1·5	20·0	10·0	Bal.	—	15·0	—	—	—	1·0	1470°F. (798°C.)

Brazing Alloys		Composition											Nominal Brazing Temperature		Super-heat Brazing Temperature	
Number	Type	C %	Si %	Mn %	Cr %	Ni %	B %	S %	P %	Zr %	Al %	Fe %	°F.	°C.	°F.	°C.
1	Ni-Cr-Si-B-C	0·76	4·60	—	13·67	Bal.	3·13	—	—	—	—	4·29	1970	1076	2060	1126
2	Ni-Cr-Si	0·17	9·91	0·81	18·95	Bal.	—	0·005	0·005	0·03	0·08	0·49	2120	1160	2210	1210
3	Ni-Cr-Si	0·03	11·25	—	19·0	Bal.	—	—	—	—	—	1·1	2150	1176	2250	1232
4	Ni-Si-B	—	4·58	—	—	Bal.	2·89	—	—	—	—	—	2070	1132	2150	1176

Cooling rate from brazing temperature was about 165°-210°F./min. (92°-117°C./min.) down to 1470°F. (798°C.), below which the rate steadily decreased.

'Irrigation', or lateral diffusion of the elements from grain-boundary regions into the grains of the base metal, can occur, as was demonstrated in some tests by the formation and precipitation of compounds of these elements within the grains of the base metal.

Incipient volume diffusion, which would occur predominantly at brazing temperature, where diffusion would be less structure-sensitive, involved elements of larger atomic radii, e.g., nickel and chromium. Diffusion of these elements into the surface grains of the base metal, by substitutional diffusion mechanisms, resulted in solid-solution formation in this region. Sometimes the solid solution produced by a diffusion reaction was unstable, and decomposed, on cooling from the brazing temperature, into a solid solution of different composition, in a eutectoid matrix.

(*b*) Deviations from the general diffusion reactions occurred in some brazing tests.

The carbon of Brazing Alloy No. 1 diffused along grain-boundary regions and lattice interstices of the surface grains in the titanium-hardened iron-base alloy and reacted with the titanium to form titanium carbide. The diffused carbon (by occupying vacancy and dislocation sites in the grain boundaries, and interstices, vacancies and dislocations in the regions of the base-metal grains), reduced the free-flow paths available to other elements of the brazing alloy, thereby retarding their diffusion into the base metal.

Superheat or brazing temperatures above the liquidus of the brazing alloy usually produced interface structures similar to those obtained at a nominal brazing temperature. The extent of these interface reactions was usually greater than those produced at nominal brazing temperatures.

Ageing at temperatures simulating maximum surface temperatures for the respective base alloys resulted in little change in the microstructural nature of the diffusion reactions, although ageing promoted these reactions slightly.

Variations in hardness across the interface regions reflected the nature and extent of the diffusion-reaction products occurring in those regions.

Ultrasonic Welding of Nickel-containing Materials

See abstract on p. 155.

Attention is drawn to the fact that many of the names of materials mentioned in The Nickel Bulletin are *Registered Trade Marks*.

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